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The Mechanism of the Dyeing of Polyester Fibres with Disperse Dyes

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Measurements of the diffusion coefficients and saturation values of seventeen disperse dyes on Dacron polyester fibre under dyeing conditions seem to indicate that the differences in diffusion coefficient between the individual dyes are mainly due, after elimination of geometrical effects, to energy effects associated with the formation of hydrogen bonds, the dye being the proton donor. The efficiencies of three different carriers were approximately the same if equimolar quantities were present inside the fibre.

Introduction

The mechanism of the dyeing of polyester fibres with disperse dyes may be regarded as understood in principle. Schuler and Remington [1] found with a few simple disperse dyes a distribution between fibre and dye liquor obeying Henry's law, i.e. the equilibrium isotherm is linear up to saturation of the fibre or water with dye. The individual dyes do not then exert much influence on one another. These findings led to the view, still held today, that a dissolution of the dye in the fibre takes place when these dyes are taken up by polyester fibres. Waters [2] investigated the saturation values and relative diffusion coefficients of different disperse dyes in polyamide, cellulose acetate, and polyester fibres. It was shown that polyester fibre takes up the largest quantities of dyes, but that the dyes diffuse most slowly in this type of fibre. The conclusion was reached that dye-binding groups must be present, although more exact details in this respect could not be given. Numerous investigations, generally of an empirical nature, have also been published on the action of carriers e.g. [3]. These experiments have made it possible to limit the choice of suitable substances as carriers. From a scientific standpoint the publications of Vickerstaff [4] and the more recent work of Schuler [5] concerning the action of carriers are very interesting. The latter work deals with investigations in non-aqueous systems, so that we cannot readily relate the results obtained to the aqueous system which is used in practice.

Apart from measurements carried out on a single dye or a few dyes at a time, there has been no exact study of the physicochemical properties, in conjunction with those which are important for dyeing, e.g. dyeing rate, saturation concentration, etc. Moreover, the action of carriers in the aqueous system has not yet been fully explained.

The aim of the work described in the present paper was to examine these questions in greater detail.

Theoretical

RATE OF DYEING

One of the main problems in the dyeing of polyesters is the low rate of dyeing. This is due to the slow diffusion of disperse dyes in the polyester fibre. Since diffusion inside the fibre is the process which governs the dyeing rate, the diffusion coefficient may be regarded as a characteristic measure of the dyeing rate. Information on the diffusion coefficient is preferable to information on the dyeing rate, because the diffusion coefficient is independent of the total quantity of dye absorbed: it merely indicates how rapidly an equilibrium is established. Moreover, the diffusion coefficient is independent of the liquor ratio and is not affected by the dispersing agent, assuming that the latter does not penetrate into the fibre. The diffusion coefficient therefore appeared to be a suitable parameter for comparing the dyeing rates of the various dyes.

DIFFUSION AT 100°C. IN ABSENCE OF A CARRIER

Direct measurements of diffusion in polyester fibres are difficult and, to ensure a reasonable degree of accuracy, require the use of fairly complicated apparatus. Such measurements were carried out on polyamide fibre by Luck [6]. B. Kramer [7] reports diffusion measurements carried out on polyamide blocks. As far as we are aware, however, absolute diffusion coefficients in polyester

material have not yet been published. To avoid the direct measurement of diffusion we followed Hill [8] in calculating the diffusion coefficient from the exhaustion curve, assuming that the fibre can be regarded as a homogeneous circular cylinder (see p. 147). In the case of Dacron polyester fibre, which was used for our investigations, this assumption may be regarded as correct.

As described in detail in the Experimental Section (p. 146), we determined the exhaustion curves of the dyes shown in Table I. At first no carrier was added. With the procedure adopted, the dyebath concentration was maintained practically constant throughout the dyeing, as is necessary if Hill's method is to be applied. The exhaustion curves themselves show that considerable differences exist in the rate of diffusion of the individual dyes. The diffusion coefficients determined from the exhaustion curves without a carrier range from $0{\cdot}033 \times 10^{-11}$cm.^{2}/sec. for dye No. 10 to $0{\cdot}62 \times 10^{-11}$cm.^{2}/sec. for dye No. 1, corresponding to half-dyeing times of 30–60 min. On the average, the diffusion coefficients for anthraquinone derivatives are higher than those for azo dyes.

Diffusion in the fibre is governed, on the one hand, by the shapes of the diffusing molecules and the intermolecular spaces in the fibre structure, i.e. by *geometrical properties*, and on the other hand by the intermolecular forces between dye and fibre, i.e. by *energy effects*. The larger a molecule in relation to the intermolecular spaces and the stronger its interaction with the fibre substance, the slower will be its diffusion rate. In our view, the polyester molecule chain can interact with dye molecules both by means of dipole, induction, and dispersion (London) forces [9] and also by the formation of hydrogen bonds. In the latter case the carbonyl group of the polyester acts as the proton acceptor. As there is no apparent proton-donating group in the polyester molecule, hydrogen bonds can be formed only with dyes which are proton donors.

To form an idea of the geometrical influences on diffusion we established a scale value for the volume of the dye molecule with the aid of true-to-scale molecular models. In each case the longest diameter of the molecular model and the two diameters perpendicular to it were measured and multiplied together. The product S of these three diameters is the volume of the circumscribed parallelepiped of the model, i.e. a size factor of the molecule. In Fig. 1 the reciprocal diffusion coefficients are plotted as ordinates against the size factors S. The hindrance to diffusion (high $1/D$ values) may in extreme cases be determined mainly by the size of the molecules, but is generally greatly influenced by energy effects also.

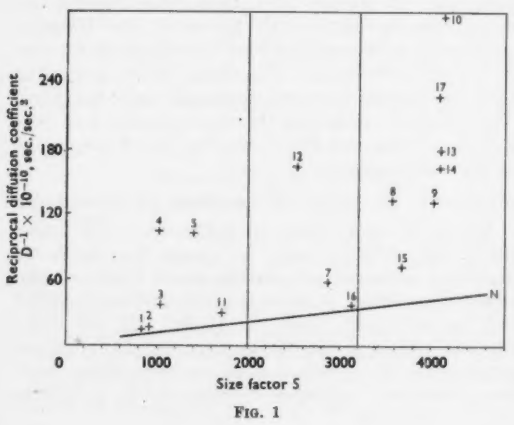


FIG. 1

Assuming that the geometrical interaction, i.e. the retarding effect on the diffusion due to geometrical properties, is a monotonic function of S, the points in Fig. 1 nearest to the abscissa axis represent dye molecules whose diffusion is hindered least by energy effects. Imaginary molecules hindered only by their size should therefore not be located at a higher level than the lowest points plotted in the diagram. In other words, they would be located below line N in Fig. 1. The fact that most of the points are located considerably above this line seems to indicate that in the range of magnitudes of the dyes examined energy effects are far more responsible for the differences in the diffusion rate than geometric properties.

In order to be able to discuss Fig. 1 in detail, the dyes plotted were divided arbitrarily into three groups. The differences in diffusion rate within the individual groups can be attributed almost entirely to energy factors, since the molecules in each of the groups do not greatly differ in their size factor S.

With the exception of a nitrodiphenylamine dye (No. 11) the first group consists entirely of anthraquinone dyes. The most rapid diffusion is shown by dyes No. 1 and 2. This can be attributed to the fact that the hydroxyl groups of these molecules, located in the *ortho* position relative to the carbonyl group, are largely involved in intramolecular hydrogen-bond formation and therefore have little tendency to interact with the fibre molecules. In products with points located at a greater distance from the line N, dyes No. 4 and 5, as well as dye No. 3, the hindrance to diffusion may be ascribed to the presence of halogen atoms, and also in part to that of additional hydroxyl and amino groups. Owing to steric hindrance the free hydroxy group in the *para* position of dye No. 11 would not be able to form hydrogen bonds with the fibre. It therefore plays only a minor rôle in hindrance to diffusion. Hydrogen bonds are formed only when the two radicals can approach very close together (approximately within ionic radii). Since the carbonyl group of the polyester is in a fixed position, the hydroxy group of the dye must be sufficiently mobile. This is the case, for example, with bishydroxyethylamino groups, but not with a hydroxy group *para* to the azo link.

The second group of dyes contains an anthraquinone dye (No. 7) and two azo dyes (No. 12 and 16). The pronounced hindrance to diffusion of dye No. 12 may be attributed to the nuclear acetamido group, and to a certain extent to the *ortho* hydroxy group, which acts as a proton donor. Dye No. 16 shows remarkably rapid diffusion, considering that

it has four groups with presumably high electric dipole moment (halogens and nitro group). The dominating influence of proton-donating groups is made apparent by comparing dyes No. 16 and 14.

Apart from two anthraquinone dyes the third group consists entirely of azo dyes. The considerable hindrance to diffusion of dye No. 14 and of the other dyes of similar structure (No. 13 and 17) is probably dye to the hydroxy groups in the hydroxyethylamino or bishydroxyethylamino groups. It can be seen from the molecular model that hydroxyethylamino groups are sterically in a position to form hydrogen bonds without difficulty.

Dye No. 10 diffuses still more slowly than the above hydroxyethylamino derivatives, this being due to the presence of a hydroxy group which may act as a proton donor and probably to the fact that the benzoic ester configuration in the dye molecule bears some resemblance to that of the fibre molecules. Therefore this dye more than others may be subject to attractive forces of the type which operate between the fibre molecules. Dye No. 15 shows only slight interaction with the fibre because its molecule has no mobile proton-donating group. The *para* hydroxy group is unable to form a hydrogen bond with the fibre molecule for steric reasons, as discussed in connection with dye No. 11.

From the above discussion it would seem that the general trends in the relation between diffusion coefficient and dye constitution can be explained on the assumption that the important factor in the interactions between dye molecule and polyester chain is the formation of hydrogen bonds, the dye being the proton donor. The total electric dipole moment of the dye molecules cannot be regarded as the source of strong interaction, as there is no correlation between dipole moment and diffusion coefficient (Table I). Also the partial electric dipole moments of separate groups in the dye molecule, such as halogen, do not seem to contribute to energy effects (dyes No. 13, 14, 16, 17). Dispersion forces (London forces) are not likely to show any influence in this connection, as they are not very specific and will therefore affect all dyes to much the same degree. The absolute effect of dispersion forces might be considerable, but differences in diffusion rate between different dyes will not arise from this cause.

DIFFUSION AT 100°C. IN PRESENCE OF A CARRIER

Exhaustion curves were determined, and hence diffusion coefficients calculated, under similar conditions but with benzoic acid and trichlorobenzene as carriers. The diffusion coefficients (Table I) are 10–100 times as high as without a carrier. It is interesting to note that the efficacy of the carriers depends largely on the nature of the dye. A certain levelling of diffusion rates takes place; i.e. dyes which diffuse slowly without a carrier are accelerated more than those which diffuse rapidly. (The efficacy of a carrier may be judged by the increase in dyeing rate obtained under definite conditions.)

Schuler[5] found that all carriers are almost equally active when present in the fibre in equimolecular concentrations. This means that the effect of a carrier depends only on the number of its molecules present in the fibre and not on their nature. The chemical structure of the carrier is important only in determining the distribution of the carrier between water and fibre. This was unexpected. It may be assumed that the action of both hydrophobic and hydrophilic carriers is based on the breaking of interchain forces in the fibre. It could, however, be imagined that the mechanism is different in the two cases: carriers containing proton-donating groups, and therefore being hydrophilic, form hydrogen bonds with the carbonyl groups of the polyester; whereas hydrophobic carriers cannot be attached to the polyester in this way, but only by dipole, induction, and dispersion forces. Attachment by means of hydrogen bonds might have a quite different loosening effect on the bonds between neighbouring polyester chains from attachment by other intermolecular forces, and this difference might lead to a fundamental or gradual difference in carrier action. Schuler showed that this is not the case.

In his experiments, however, Schuler used *iso*octane as dyeing medium, and found that water itself is an excellent carrier. It was now interesting to see whether Schuler's findings applied also to dyeing from an aqueous bath. As is shown in Table I, in most cases the efficacy of benzoic acid and trichlorobenzene in the concentrations applied, 20 and 1 g./litre respectively, was roughly the same on the average, trichlorobenzene being slightly more effective.

To find the concentration of the carriers actually inside the fibre we determined the distribution curves between water and fibre for benzoic acid and trichlorobenzene (Fig. 2). These show that when 20 g. of benzoic acid per litre of solution is used, approx. 0·35 millimole of benzoic acid is absorbed per gram of the fibre. The corresponding concentration of trichlorobenzene is 0·55 m-mole/g. when 1 g. of trichlorobenzene per litre is added to the liquor. As previously stated, both these carriers accelerate diffusion to approximately the same extent when used in the above proportions. The number of trichlorobenzene molecules is in fact of the same order of magnitude as that of the benzoic acid molecules, and it therefore appears that Schuler's findings apply also to practical dyeing conditions from aqueous baths. This view is supported by experiments with another compound used as a carrier, an aromatic ester of a carboxylic acid*. This compound was also tested for its activity, in relation to the quantity used. The same effect as in the case of trichlorobenzene was found when 0·55 m-mole of the ester is dissolved per gram of the fibre, i.e. when it is present in the same molecular concentration as trichlorobenzene. These comparisons were carried out with dye No. 2. In connection with the determination of the carrier isotherms, the water content of the carrier-containing test specimens was examined (by the Karl Fischer method). Approximately the same water content as in the samples without a carrier was found (about 0·4%).

* Levegal PT (FBy).

TABLE I

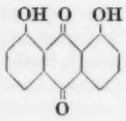
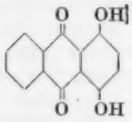
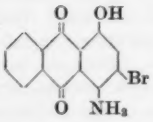
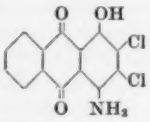
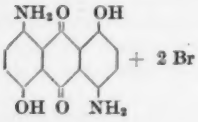
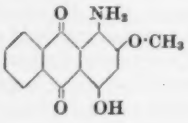
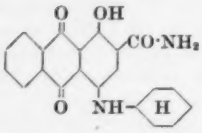
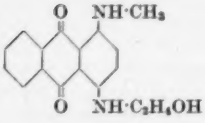
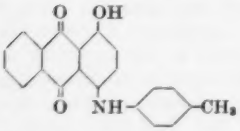
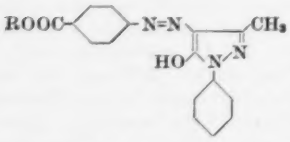
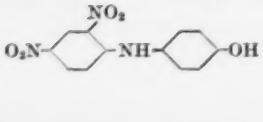
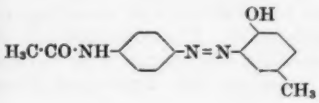
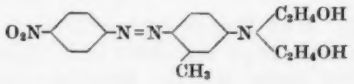
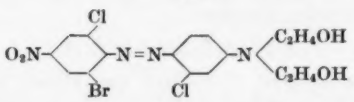
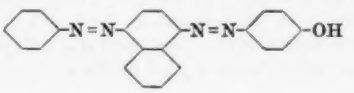
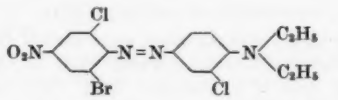
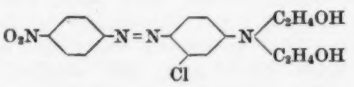
Dye No.	Constitution	*Colour Index* No.	Mol. Wt.	Dipole Moment	Saturation Values (mg. pure dye/g. fibre)			Diffusion Coefficient ($cm.^2/sec.) \cdot 10^{11}$		
					Without Carrier	Benzoic Acid	Trichloro-benzene	Without Carrier	Benzoic Acid	Trichloro-benzene
1		—	240	—	41	36	48	0·62	13 ±1	18 ±2
2		—	240	2·6	34	33	39	0·6	23 ±9	14
3		—	318	3·5	15	11	15	0·28	9·5	33 ±3
4		—	308	3·2	7·6	5·4	6·9	0·10 ±0·01	4·5	23 ±9
5		—	428	—	28	24	22	0·1	3·1	9·5 ±1
6		C.I. 60755 C.I. Disperse Red 4	269	3·0	—	24·5	—	—	7·7 ±0·8	—
7		C.I. 62050 C.I. Disperse Blue 6	364	5·4	25	54·5	59	0·17 ±0·03	1·8 ±0·2	1·5 ±0·3
8		C.I. 61505 C.I. Disperse Blue 3	296	—	62	38	—	0·07 ±0·02	3·0 ±0·6	—
9		—	329	—	17·5	15	—	0·07 ±0·02	2·8 ±0·3	1·1
10		—	336	—	63	69	59	0·03 ±0·01	1·2 ±0·1	1·1 ±0·1
11		C.I. 10345 C.I. Disperse Yellow 1	275	4·3	34	20	36	0·34	6·2	22 ±4

TABLE I— *continued*

Dye No.	Constitution	*Colour Index* No.	Mol. Wt.	Dipole Moment	Saturation Values (mg. pure dye/g. fibre) Without Carrier	Benzole Acid	Trichlorobenzene	Diffusion Coefficient ($cm.^2/sec.) \cdot 10^{11}$ Without Carrier	Benzole Acid	Trichlorobenzene
12		C.I. 11855 C.I. Disperse Yellow 3	260	3·6	37	30	25	0·06	2·3	18 ±2
13		C.I. 11210 C.I. Disperse Red 17	344	6·3	25	—	32	0·05	—	5
14		—	478	—	54	43	75 ±10	0·06	2·3	2·3 ±1
15		C.I. 26080 C.I. Disperse Orange 13	352	—	20	25	—	0·13	1·8 ±0·2	—
16		—	446	—	10·3	25	50 ±10	0·26 ±0·03	1·1 ±0·1	—
17		C.I. 11150 C.I. Disperse Red 7	365	4·3	24·4	—	31·5	0·043	—	0·35 ±0·04

To summarise, it may be stated that the effect of the carrier in the fibre does not depend directly on the type used but on the number of molecules present in the fibre. Thus, the steeper the distribution curve of a substance, i.e. the more hydrophobic this substance is, the more suitable it will be as a carrier. In practice, however, the carrier is required to satisfy other conditions, particularly good dispersion properties and minimum volatility, quite apart from price considerations and possible toxicity. To ensure satisfactory dispersion and to reduce volatility, it is advisable to incorporate hydroxy or other hydrophilic groups in the carrier. Thus, it is not really possible to use completely hydrophobic substances, but a compromise must be effected between the hydrophobic character and other properties of a substance.

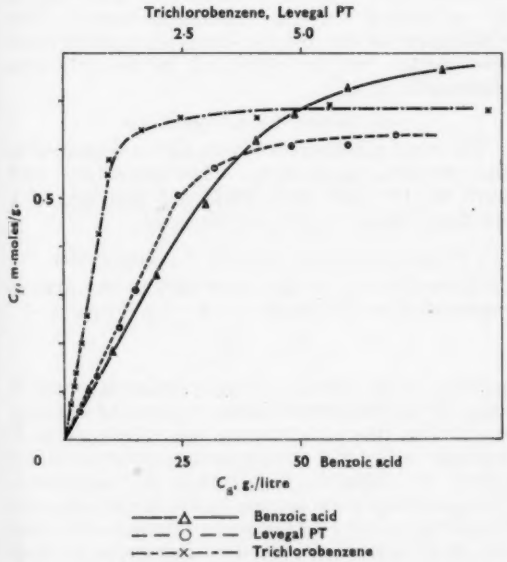


FIG. 2— Equilibrium Isotherms of Carriers at 100°C. (C_f and C_s = Equilibrium concentrations on the fibre and in the solution respectively)

DIFFUSION AS A FUNCTION OF TEMPERATURE

It is well known that a carrier is not required for the dyeing of polyesters at temperatures exceeding 100°C. (high-temperature dyeing process). This is due to the fact that the diffusion coefficient increases with rising temperature. The extent of this increase was found by measuring exhaustion curves at different temperatures. The experiments were carried out in a Dyeometer with dye No. 11 because of its relatively high solubility in water (450 mg./litre at 100°C.), since in the Dyeometer only clear solutions can be measured. From these curves the diffusion coefficients were calculated by a suitable method (see Experimental Section).

It was to be expected that the dependence on temperature of these D_T values would be in accordance with the relation—

$$D_T = D_0 e^{-E/RT}$$

where D_0 is a constant, E the molar energy of

activation, T the absolute temperature, and R the general gas constant. A straight line must therefore be obtained when $\log D_T$ is plotted against $1/T$. The energy of activation E can then be calculated from the slope of this straight line. In the present case the energy of activation proved to be $E = 34 \pm 1$ kcal./mole. Strictly speaking, this value applies only to this particular dye and fibre type. It has been found, however, that the activation energies of dyeings carried out on a definite fibre type with different dyes of the same group show little difference provided that the dyeing mechanism is the same. This value may therefore be regarded as a measure of the activation energy for the dyeing of Dacron with disperse dyes. Fibres with different degrees of stretch and of different origins may differ in their activation energies (D. Patterson, *personal communication*). The activation energy found is higher than for dyeings on wool, for dyeings with disperse dyes on Perlon (20–25 kcal./mole), and for dyeings with direct dyes on cellulosic fibres (approx. 15 kcal. per mole)[10], but lower than for basic dyes on Dralon (72 kcal./mole)[11]. The dyeing rate on polyester fibres (particularly Dacron) is therefore less dependent on temperature than that on polyacrylonitrile fibres (particularly Dralon).

Experiments carried out up to the present have indicated that the energy of activation seems to fall when dyeing with a carrier. This may be due to the temperature dependence of the distribution of the carrier, i.e. to less carrier being present in the fibre at higher temperatures. Further investigation of this point is necessary.

Experimental

MATERIALS

Commercial Dacron hank yarn was used. The fibre diameter was 18·5 μ.; the metric yarn count 60/2. The material was prescoured for 20 min. at the boil with 1 g. Levapon CA and 1 g. trisodium phosphate per litre.

Chemicals were of technical purity. The trichlorobenzene was mainly the 1:2:4 isomer. Distilled water was used.

The dyes were of commercial quality and not purified. It was assumed that other substances in the dyes would not penetrate the fibre and therefore not directly interfere with diffusion.

It is possible that a dispersing agent increases the absolute dyeing rate, because more dye particles small enough to enter the fibre are present. This must, however, result in a corresponding rise in the saturation value. Since the calculation of the diffusion coefficient involves only the ratio of dye absorbed and saturation value, the influence of a dispersing agent is eliminated, provided that the amount of dye absorbed and the saturation value are changed by the same factor.

EXHAUSTION CURVES

(*a*) *Without Carrier*

Ten 0·2-g. portions of Dacron fibre were boiled under reflux in 1·8 litres of dye liquor containing 1·8 g. of dye. 0·2 g. of fibre was withdrawn after 10, 20, 40, 80, 160, 240, and 360 min. and 24 and 48 hr. respectively. This was continued until equilibrium was established, for which a maximum of 96 hr. was required. After withdrawal the samples were rinsed, treated with 2 g. soda and 2 g. hydrosulphite per litre at 70°c. for 20 min. to remove any loose dye, and then dried. 100 mg. of the sample was subsequently extracted with hot chlorobenzene, and the extract was made up to 100 ml. with chlorobenzene. The concentration of the dye solution was then determined with the aid of a calibration curve on the Eppendorf filter photometer.

(*b*) *With Benzoic Acid as Carrier*

Benzoic acid (36 g.) was added to the liquor set as described under (*a*), the procedure adopted being otherwise the same.

(*c*) *With Trichlorobenzene as Carrier*

Trichlorobenzene (1 g./litre) was added to the liquor set as described under (*a*). To prevent any decomposition of the emulsion during boiling, a reflux condenser was not used, but the experiment was carried out in sealed glass flasks at 100°c.

ISOTHERM OF BENZOIC ACID

Dacron (2 g.) was in each case boiled for 7 hr. under reflux in 320 ml. of water with 1·6, 3·2, 6·4, 9·6, 12·8, and 16 g. benzoic acid. The samples were then quickly rinsed with water and subsequently treated for a short time in ice-cold acetone at −15°c. to remove water and surface deposits, dried for 15 min., and weighed. The quantities of carrier in the samples were found from the differences in weight.

ISOTHERM OF TRICHLOROBENZENE

Dacron (2 g.) was in each case treated in 320 ml. of water for 7 hr. in sealed glass flasks at 100°c. with 160, 320, 640, 960, 1920, and 2560 mg. trichlorobenzene (and in each case 0·1% Emulsifier W calculated on the trichlorobenzene). The preparation of the test specimens for measurement was carried out as described in the previous paragraph.

ISOTHERM OF LEVEGAL PT

The same procedure was adopted as described in the previous paragraph. The quantities used were 84, 128, 256, 512, 1024, and 2048 mg. of a solution containing 83% of the ester.

CALCULATION OF DIFFUSION COEFFICIENT

If the diffusion of dye molecules in the fibre is represented in the usual way by the equation—

$$\frac{ds}{dt} = -D\frac{dc}{dx}$$

where s is the quantity of dye diffusing per unit area, dc/dx the concentration gradient of the dye, and t the time, experience has shown that D depends on the dye concentration, and is therefore called the *diffusion coefficient* (not "constant"). This dependence on concentration is due either to the diffusion being determined not by concentration but by the activity of the dye (Patterson, *personal communication*), or to the diffusion taking place along channels on the walls of which the dye is retained. The last-mentioned factor appears to

apply in particular to cellulose fibres. If the diffusion coefficient is calculated from the total quantity of dye taken up in each case a mean value, the "apparent diffusion coefficient", is obtained which remains practically constant during the entire dyeing process. The diffusion coefficient is in this paper always understood to be this mean value.

To calculate D from our measurements we first used Hill's formula [8] for diffusion into long cylinders, the concentration in the dyebath remaining unchanged—

$$\frac{C_t}{C_\infty} = 1 - 0{\cdot}692(e^{-5{\cdot}78b} + 0{\cdot}190e^{-30{\cdot}5b} \ldots)$$

where C_t is the quantity of dye taken up by the material in time t and C_∞ the quantity taken up after a very long time; $b = Dt/r^2$, where r is the radius of the cylinders.

From the measurements we calculated C_t/C_∞ and determined by means of a graph (showing Dt/r^2 as a function of C_t/C_∞) the corresponding values of Dt/r^2. The time t was known, and the mean radius of the fibres was found under the microscope to be $r = 9{\cdot}25 \pm 0{\cdot}3\ \mu$.; D could therefore be calculated.

Apart from occasional deviations in the case of very short measuring times, D was fairly constant for all C_t/C_∞ values of a dyeing, with a mean deviation of about 10%. A slight systematic fall or rise often occurred during dyeing. The stated values are average time values, the accuracy of which differs considerably (unless otherwise stated, the error is about 5%).

In evaluating the measurements in the high-temperature Dyeometer, in which the dye concentration decreases during dyeing, an extension of Hill's formula due to Crank [12] was used.

DETERMINATION OF EXHAUSTION CURVES IN THE HIGH-TEMPERATURE DYEOMETER

A dispersion (300 c.c.) containing 125 mg. of dye No. 11 was heated in four experiments to 100°, 116°, 124°, and 138·5°C., clear solutions being obtained. The material, i.e. 10 g. Dacron hank yarn of the same type as used for the previous measurements, was entered, and the change with time in the exhaustion of the liquor was measured with a recording photometer. The quantity of dye taken up was calculated from the values obtained. The linearity between exhaustion and concentration was checked.

* * *

The authors wish to express their thanks to Dr. P. Rabe for recommending this study as well as for his valuable suggestions in the course of the work.

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COMMUNICATION

Flash Photolysis of Vat Dyes

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A brief outline is given of the methods which have been used to investigate the processes occurring when fading and phototendering take place. The results obtained using model systems and the technique of flash photolysis are extended in this work to the more complex problem of commercial vat dyes in solution. It is shown that closely similar species exist in these latter solutions, namely the semiquinone radical and radical-ion, and that the reactions taking place on light absorption, as well as the behaviour of the radicals under various conditions, are similar to those obtained for simple quinones. Active tenderers are found to yield, on illumination, greater amounts of radicals with longer lifetimes than inactive dyes. These results are considered to be a justification for using the above methods to help understand the practical case of vat dyes on fibres.

INTRODUCTION

The study of the fading of dyes and of their phototendering of textile materials has occupied a considerable fraction of the time and effort spent on research in the industry. Numerous workers have surveyed the results of technological and fundamental work carried out on all classes of dyes, on many kinds of fibre, and under vastly different conditions [1–3]. Much of the work has been exceedingly valuable in defining the conditions under which the damaging actions take place, in correlating the degree of this attack with structure, and in suggesting specific methods of prevention [4–8].

However, it has become increasingly clear that, in order to arrive at a thorough understanding of the complexities of the problems, considerable experimental simplification must be made. Amongst these complexities are the structure of the dye molecule (molecular weights of the order of 800 are not uncommon); the fibre structure, which is very involved whether considered from a physical or a chemical viewpoint; and the many variables that can affect the final amount of fading and tendering. By simplifying conditions in the

manner suggested, a gradual improvement in the understanding of detailed mechanisms has been obtained [9–11].

It is because of the good light fastness of vat dyes as a class that the phototendering of cellulosic fibres they cause has received such a great deal of attention. Up to the present time two types of experiment have been carried out in order to unravel the processes involved. In the first method, which has possibly yielded more practical information, samples of a given type of fabric, dyed with various commercial vat dyes, are subjected to visible light under strictly controlled conditions. Subsequent analysis of any detectable product, or of any colour changes, is used to help postulate a mechanism. The second method entails a longer, though more fundamental, approach, in which a so called "model system" is set up. Here, simple dyes which are considered to typify the more complex ones are chosen, e.g. simple anthraquinones for the vat dyes. These, because of the difficulties of studying solid-state as compared with liquid(or gas)-state reactions, are dissolved in liquids having chemical properties related to those of the fibre, e.g. ethanol or ether for cellulose.

The work of Bolland and Cooper on the photosensitised oxidation of ethanol by anthraquinones is a recent example of the use of the latter technique [12]. From a detailed study of the products and oxygen uptake obtained when sodium anthraquinone-2:6-disulphonate dissolved in aqueous ethanol is irradiated by light in the blue and near ultraviolet regions of the spectrum, they proposed a reaction scheme which explained quantitatively the observed processes. In common with previous workers in this field they suggested a radical mechanism involving in particular a semiquinone radical (QH•) produced by the reaction of an excited sensitiser molecule (Q*) with the substrate (RH). The first two steps of the reaction, which are the main concern of the present paper, can be written—

$$Q + \text{Light} \longrightarrow Q^* \qquad \text{(i)}$$

$$Q^* + RH \longrightarrow QH\bullet + R\bullet \qquad \text{(ii)}$$

There is no reaction of Q* with O_2 [13, 14], and the subsequent oxidation processes involve reactions of QH• with O_2, and of R• with both Q and oxygen.

If these primary processes also apply to vat-dyed fibres, then it is clear that, in order to prevent the fading and phototendering, the course of reactions (i) and (ii) should be known in the greatest possible detail, so that they might be changed (see also Wells [14]). Work of this nature is proceeding in these laboratories.

However, in the above kind of study no direct evidence is obtained regarding the nature of the excited state, which can be either singlet or triplet (see Fig. 1). In general all the electrons in a molecule (Q) are paired together with opposite spins (↑↓)— the molecule is in a spectroscopic *singlet* state— and absorption of light results in the exciting of one electron to a state of higher energy such as S*, without, however, change of spin direction, so that this excited state also is singlet. There is then a small, but finite probability that the molecule in such an excited state might pass over, by a non-radiative transition, into another set of excited states the lowest of which is shown as T— the spectroscopic *triplet* state— in which the spin of the excited electron has been reversed and the molecule as a whole now has two (unpaired) electrons with parallel spins (↑↑ or ↓↓). Light absorption can also take place when the molecule is in the T state, resulting in a triplet absorption spectrum entirely analogous to the normal singlet absorption spectrum. This triplet state, populated either as described above, or by the very weak Q → T "forbidden" absorption process, is metastable with a much longer lifetime than the excited singlet state, and probably exists as a diradical with the two unpaired electrons in different parts of the molecule. The possibility that the reactive state Q* might be triplet has been considered by many authors (cf. Bowen and others at the 1949 Harrogate Symposium on *Photochemistry in relation to Textiles* [1]), and since then a great deal of work has been carried out on processes supposed to involve the triplet state [15–17]. Of direct interest is the work of Oster *et al.* [18], who from a study of the fading and luminescence of dyes in aqueous solution (containing also water-soluble polymers) have obtained convincing evidence that the active state is long lived (approx. 10^{-5} sec.) and is probably the triplet state. However, it should be noted that if the transition responsible for the absorption of light by the dyes used in Oster's work is a weak ($n \rightarrow \pi^*$) transition, then the lifetime of the corresponding excited singlet state (S*) resulting from such an absorption is probably as long as 10^{-6} sec., and might be confused with a triplet lifetime.

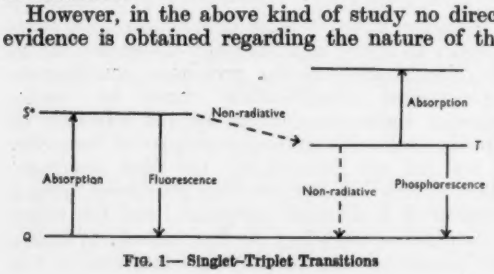

FIG. 1— Singlet–Triplet Transitions

Direct evidence from the production, by light, of molecules in the triplet state with lifetimes between 10^{-5} and 10^{-2} sec. has been obtained recently by the technique of flash photolysis devised by Porter [19]. From a study of a large variety of molecules in solution Porter and Windsor concluded [20] that triplet state formation "is a phenomenon of very general occurrence in a wide variety of compounds" and is "probably one of the most important primary processes involved in the photochemical behaviour of many organic compounds and in particular of dyestuffs, where the industrially important phenomena of fading and phototendering of dyed fabrics may well have an explanation in terms of the phosphorescent state". Following this work, Bridge and Porter [21], using the flash technique, have recently obtained evidence that simple anthraquinones and naphthaquinones, as well as various methyl-substituted benzoquinones, especially tetramethyl-*p*-benzoquinone (duroquinone), on irradiation give rise to semiquinone radicals and to

triplets. In particular, it was shown that those anthraquinones that are good sensitisers for the photochemical oxidation of alcohol [13] on flashing gave large concentrations of relatively long lived radicals, whereas non-sensitisers gave little if any such radical absorption, and this of short life. Also, contrary to expectation, it was found for duroquinone in the viscous, inert solvent liquid paraffin (chosen to prolong the lifetime of any transients, e.g. radical, radical ion, excited sensitiser) that the triplet does not react significantly with the substrate to produce the semiquinone radical (QH•), but probably the excited singlet state (of lifetime approx. 2×10^{-6} sec.) does so. Evidence was also obtained that in ethanol the hydrogen-abstraction reaction (ii) occurs and not the alternative electron-transfer reaction—

$$Q^* + RH \rightarrow Q^{-}\cdot + RH^{+}$$

in agreement with the results of Wells [22].

The work reported below is an attempt to detect the same processes and radicals for commercial vat dyes in solution.

EXPERIMENTAL

Experimental Technique

The general principles and details of the technique of flash photolysis for the study of fast chemical reactions were described originally by Porter [19]. The paper by Bridge and Porter [21] gives details of the particular arrangement used in this work. Briefly, an intense 20-μsec. "photolysis" flash of visible and ultraviolet radiation P (Fig. 2)

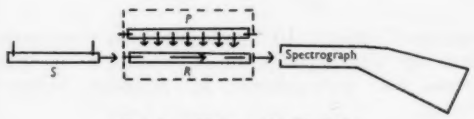

FIG. 2A— Photographic Method

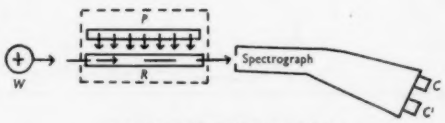

FIG. 2B— Photoelectric Method

falls on the dye solution in the reaction vessel R and initiates the photochemical production of ("coloured") radicals. (The photolysis flash emits a continuum of light from the near infrared through the visible and ultraviolet region of the spectrum down to the transmission limit of the air and the quartz— ca. 200 mμ.) These radicals are subsequently detected by allowing the white light from a less intense "spectroscopic" flash S of similar duration to pass through the solution and into a spectrograph, where the absorption spectrum of the products can be recorded on a photographic plate (see Fig. 2A). The delay between the two flashes is preset to any required value down to approx. 30 μsec. (3×10^{-5} sec.). Having established the wavelength of a transient by this method, its build-up and decay can then be studied quantitatively by replacing the second "spectroscopic" flash system with a tungsten (or xenon) lamp W and photomultiplier arrangement C, C' (Fig. 2B).

Materials

All the experiments reported below were carried out in purified ethanol varying in water concentration between 4 and 50%. Mainly because of the exploratory nature of this work, the dyes were not purified, but instead, filtered saturated ethanol solutions of the dyes were prepared as stock and then diluted as necessary. The concentrations used, estimated from a knowledge of their spectra [23], varied between 10^{-5} and 10^{-4} M. A further reason for not purifying the dyes was that the various additives present in the commercial samples— e.g. sodium chloride, sodium sulphate, dispersing agents— might possibly have influenced their photochemical properties. It was anticipated that, if no transient species could be detected, then purification would be carried out. The Caledon dyes were samples kindly supplied by Imperial Chemical Industries Ltd., and the Cibanone dyes were kindly supplied by the Clayton Aniline Co. Ltd. Unless otherwise stated, all reagents were of AnalaR grade, used without further purification.

Procedure

In order to compare results with previous work on simpler anthraquinones [21], the dye solutions were thoroughly degassed by shaking, freezing, and pumping. The concentration was always adjusted to give optimum light absorption, since too low a concentration would cause insufficient light absorption for photolysis, and too high often produced absorption obscuring the region of the expected transient. In all cases the stock solutions were diluted to approx. 10^{-5}–10^{-4} M. (see above).

In an experiment, the 22 cm. long quartz reaction vessel containing the degassed dye solution was put in the reaction chamber (shown dotted in Fig. 2) next to the flash tube P. First of all the spectrum of the solution was recorded on the spectrograph plate by flashing S (this caused no appreciable photodecomposition), and then the transients were recorded as described previously. Fig. 3 and 4 give typical results. (The lines in the spectra are due to the xenon flash and not to the transients.)

To help identify these recorded transients, microdensitometer traces of the plates were made using a Hilger non-recording instrument. Normally this procedure gave directly a very clear picture of the spectral changes occurring. However, in some cases a marked permanent photochemical change was produced on flashing, with an absorption spectrum overlapping that of the transients, and some attempt at correction had to be made even though this gave only approximate values. This was done by measuring the initial and final spectrum (S only) and then, using linear interpolation, subtracting from the appropriate delayed spectrum. The spectra given below have been corrected in this way, and the ordinate d_s is the optical density in the 22-cm. cell assuming linear

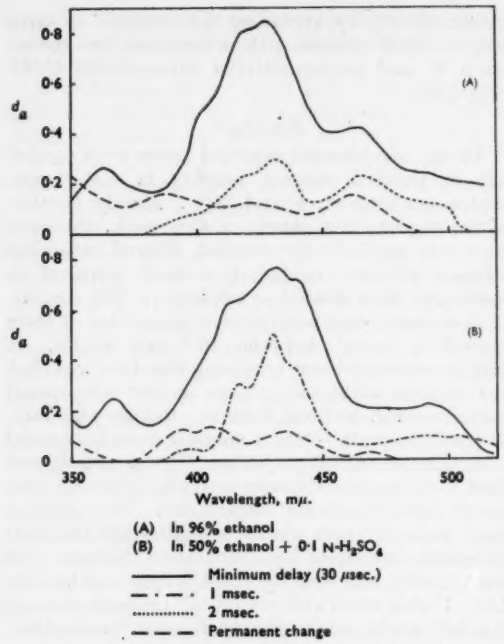

FIG. 5— Active Vat-dye Transients (Caledon Yellow 5G)

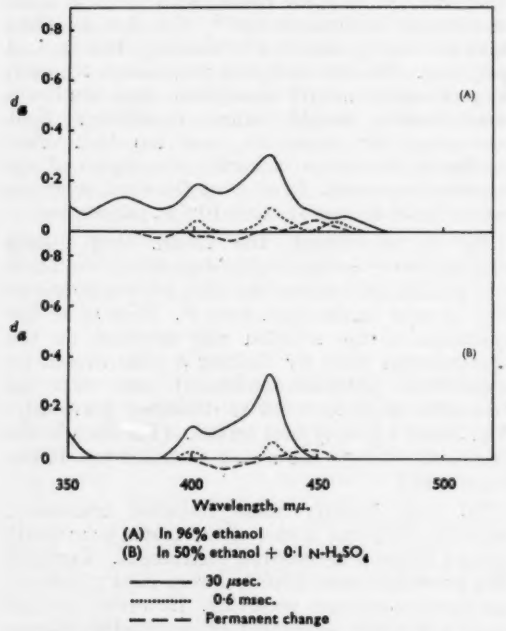

FIG. 6— Inactive Vat-dye Transients (Caledon Yellow GN)

plate characteristics ($\gamma = 1$). Fig. 5 gives typical results.

In some cases a negative transient or permanent change was observed, corresponding to regions of the spectrum in which the product absorbs less than the original dye.

In order to measure the decay of a radical accurately the arrangement of Fig. 2B was used with a photomultiplier set at the radical absorption peak and its output lead to a cathode-ray oscillograph; one flash from P then caused the whole course of the build-up and decay of the species concerned to be displayed on the cathode-ray tube, where it was photographed on 35-mm. film.

Results

In contrast to the spectra obtained when simple quinone sensitisers and non-sensitisers were flashed, the difference in behaviour between active and inactive commercial vat dyes was not quite so striking. However, as Fig. 3–6 show, there are definite differences. (Fig. 3 and 5 are for active Caledon Yellow 5G, and Fig. 4 and 6 are for inactive Caledon Yellow G and Caledon Yellow GN respectively— for details of the activity of these vat dyes see e.g. references [2, 4].) The main differences are the greater concentration and longer lifetime of the transient formed from the active vat, and the considerably greater permanent change produced on flashing such a dye. Interesting and important though these differences are, the main object of the work was to establish whether the same radical types are produced from the present complex dyes as from the simpler ones.

To study this problem, use was made of the previously described technique of adding acid or alkali to the ethanol solutions of the dye and observing the transients produced on flashing. It was expected that, if the observed transients were semiquinone radicals, the spectra would differ in the two cases, because the radical (QH•) and the radical-ion (Q^{-}•) exist always in equilibrium —

$$QH\bullet \rightleftharpoons Q^{-}\bullet + H^{+}$$

A value of approx. 10^{-6} mole/litre was previously found for the equilibrium constant K of this process for duroquinone in aqueous ethanol solution.

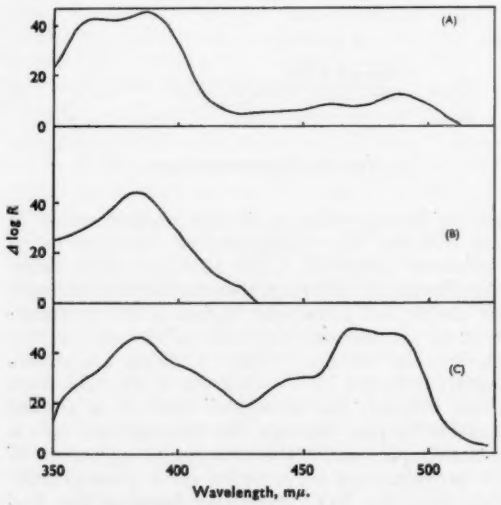

FIG. 7— 2-Methylanthraquinone Transients

Fig. 5B shows the result of adding 0·1 N. sulphuric acid. The band at 460–470 mμ. in ethanol is almost completely absent when acid is added, but the main band at 430 mμ. is not greatly altered. This behaviour is remarkably similar to that found for the simpler substituted anthraquinones [21]. For example, in the case of 2-methylanthraquinone, of the two main bands found in ethanol at approx. 390 and 460–490 mμ. (see Fig. 7A) the higher one— there clearly identified as the semiquinone radical-ion— disappears on the addition of acid (Fig. 7B), just as in the present case. Further, when alkali is added to the 2-methyl compound (Fig. 7C), the high-wavelength band is greatly intensified because of the increased amount of radical-ion. (The low-wavelength band changes little because the radical-ion also absorbs in this region.) In addition, the radical-ion, once formed by a flash, has a lifetime measured in minutes because of its great stability under alkaline conditions. However, in the present case, this latter effect, which was apparent to the eye as a very intense orange colour produced on flashing the alkaline solution, could not be measured because of the very large absorption and because of the rapid attack of the alkali on the quinone to produce obscuring coloured products. (A similar effect was noted for most of the methylquinones previously tried.)

These results indicate that irradiation of commercial vat dyes under the above conditions produces exactly the same radical types as that of the simpler quinones, namely the semiquinone radical and radical-ion.

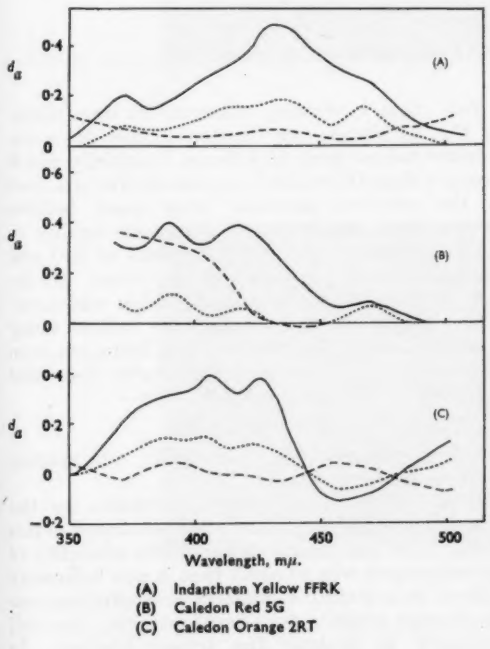

FIG. 8— Photolysis of Active Vat Dyes in 96% Ethanol

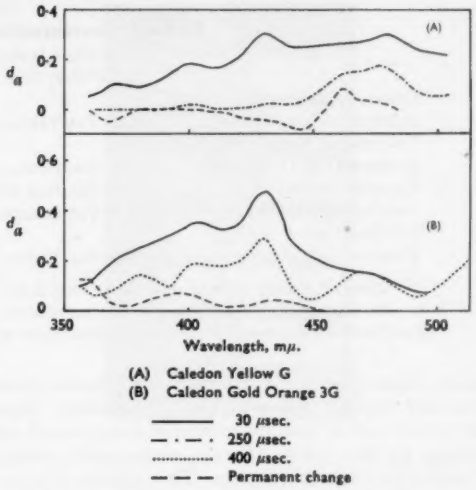

FIG. 9— Photolysis of Inactive Vat Dyes in 96% Ethanol

Additional confirmatory results were obtained as follows. First, the effect on the weaker transients of the inactive dye of adding acid is shown in Fig. 6B, where again the small $Q^{-}\bullet$ peak at 460 mμ. is weakened and the QH• peak slightly enhanced. Next, Fig. 8A–C show the semiquinone radical and radical-ion as well as the permanent change (observed after approx. 1-min. delay) formed on flashing the other active vat dyes— Indanthren Yellow FFRK, Caledon Red 5G, and Caledon Orange 2RT— in ethanol. Lastly Fig. 9A and 9B show once again the weaker transients and almost complete absence of permanent change for the inactive dyes Caledon Yellow G and Caledon Gold Orange 3G.

It is interesting to compare the amount of semiquinone radical produced on flashing at minimum delay (30 μsec.) with the activity of the dye in question. In the first column of Table I the values of d_a (the approximate optical density at the absorption maximum of the radical produced by the first flash) are given, and in the subsequent columns the activities of the various compounds as measured by various authors are shown.

This very crude comparison does show a general correlation with previous findings, large radical concentrations occurring for very active dyes. For the less active dyes the difference is not very great. In the case of Cibanone Yellow R (which is one of the most active tenderers known and is at the top of the table), the absorption produced by the first flash was so large that it could not be measured. Moran and Stonehill [8], who used the extent of radical-initiated polymerisation as a measure of activity, found that Caledon Yellow 5G produced a slight amount of polymer precipitation on irradiation, Indanthren Yellow FFRK and Caledon Gold Orange 3G a negligible amount, but, contrary to the general trend, Caledon Yellow GN produced a dense precipitate.

Other observations were made during the course of the experiments which substantiate the above assignments. It was observed that all the active vat dye solutions after flashing, as well as being deep

TABLE I

Radical Concentration, and Activity of Dyes

Dye	*Colour Index* Designation	d_s	Tendering Activity* I	II	III
Cibanone Yellow R	—	>2	52	1	26
Caledon Yellow 5G	C.I. Vat Yellow 2	0·8	46	–	14
Indanthren Yellow FFRK	—	0·5	39	1–2	10·4
Caledon Gold Orange 3G	C.I. Vat Orange 15	0·5	17	4	5·5
Caledon Red 5G	C.I. Vat Red 42	0·4	15	–	–
Caledon Orange 2RT	C.I. Vat Orange 2	0·4	25	–	4·5
Caledon Yellow G	—	0·3	26	–	–
Caledon Yellow GN	C.I. Vat Yellow 1	0·3	–	–	4·6

* I— Egerton[24], fluidity increases on cotton at 100% R.H.
II— Fox[4], using Harrison's silver test to measure cellulose degradation (1 = Highly active, 1–2 = Active, 4 = Inactive)
III— Bamford and Dewar[25], fluidity increases on viscose rayon.

yellow, were fluorescent, whereas the inactive vats were only slightly yellow and not fluorescent. Also this yellow colour and fluorescence disappeared on opening up the cell to the air, presumably owing to oxidation back to quinone. This agrees with the observations made on the simpler quinones that the resulting product in absence of air is the highly fluorescent quinol, formed by either—

$$2QH\bullet \rightarrow Q + QH_2$$

or possibly—

$$QH\bullet + RH \rightarrow QH_2 + R\bullet$$

In the case of active vat dyes it was found that the radical concentration was reduced considerably after several flashes because of the removal of the quinone, whereas this concentration was unaffected with inactive vat dyes.

To demonstrate more clearly the difference between active and inactive dyes and also to determine the lifetimes of the radicals produced, the photomultiplier technique was used to study an active vat dye, Caledon Yellow 5G, and an inactive one, Caledon Yellow GN. In Fig. 10 and 11 deflections upwards correspond to increasing optical density, i.e. increasing concentration of the radical; the full light deflection was approximately the same for both. For the active dye (Fig. 10) the wavelength (450 mμ.) was chosen to give the maximum possible difference in absorption between the short lived semiquinone radical, present at the beginning of the trace, and the permanent change produced, apparent at the end of this trace. (The absorption corresponding to the permanent change is presumably due to disappearance of the dye and appearance of products which absorb light in this region.) It was not possible to separate the two any better than this. For the inactive dye (Fig. 11) the wavelength (435 mμ.) was simply that of maximum absorption. It can be seen that there was less transient. Also, because there was no permanent change it was possible to determine the order of the radical decay by calculating the optical density d from the film and then plotting d, log d, or $1/d$ against time. Fig. 12 shows that the decay is of the first order (with rate constant $k = 3{\cdot}3 \times 10^3$ sec.$^{-1}$) and not of zero or second order. In the case of duroquinone, the decay under similar conditions was of the second order; at the present time the reason for this difference is not fully understood.

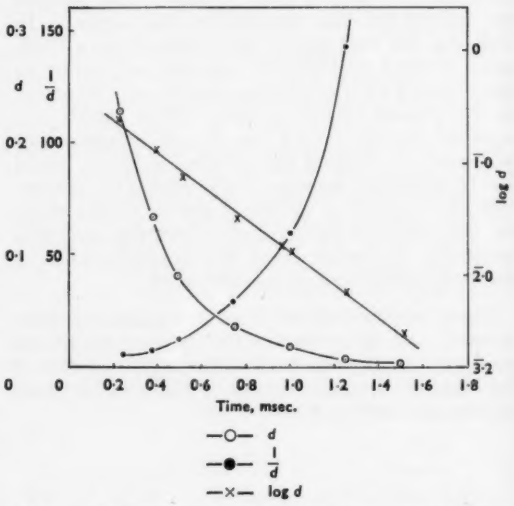

FIG. 12— Caledon Yellow GN in 96% Ethanol (d measured at 435 mμ.)

Two other interesting observations were made. As Fig. 5A shows, the Caledon Yellow 5G semiquinone radical peak at 430 mμ. is initially much stronger than the radical-ion peak at 470 mμ., but as the reaction proceeds these peak heights become more nearly equal. The same applies to the Indanthren Yellow FFRK peaks at 430 and 480 mμ. (Fig. 8A) and possibly to others. In the case of duroquinone, a similar change was interpreted as due to the semiquinone radical being produced initially by abstraction of hydrogen from the solvent, at a concentration greater than that necessary to satisfy the equilibrium—

$$QH\bullet \rightleftharpoons Q^{-}\bullet + H^{+}$$

(at the particular pH), followed by the gradual attainment of equilibrium.

It should be noted that no evidence for the production of the dye triplet state was found in this work. This was mainly because the solubility of the substances was so small that it was extremely difficult to introduce a sufficient concentration into the viscous medium (e.g. liquid paraffin, glycerol) necessary to prolong the triplet lifetime. In addition, experiments in these laboratories have shown that no good sensitisers of the simple substituted anthraquinone type (i.e. active dyes)

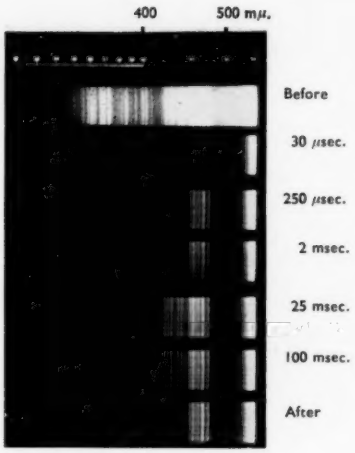

FIG. 3— Active Vat Dye (Caledon Yellow 5G)

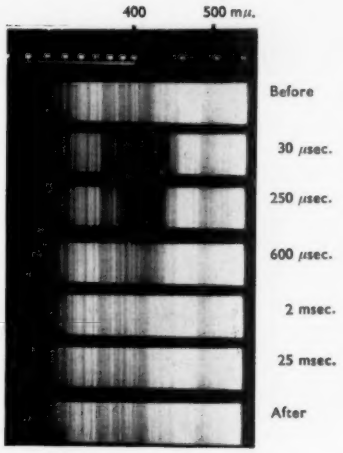

FIG. 4— Inactive Vat Dye (Caledon Yellow GN)

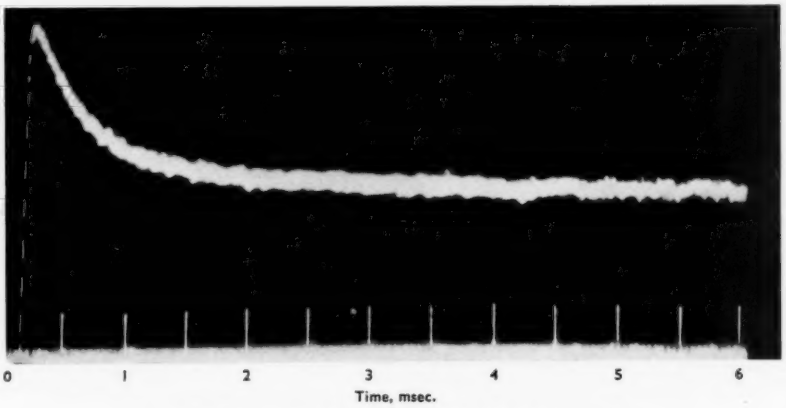

FIG. 10— Active Vat Dye (Caledon Yellow 5G) at 450 mμ.

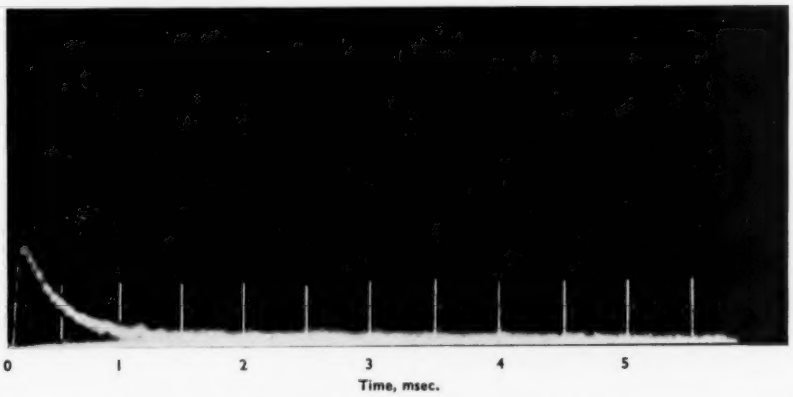

FIG. 11— Inactive Vat Dye (Caledon Yellow GN) at 435 mμ.

yield an observable triplet state on flashing at room temperature in viscous solvents. This is probably due to the rapid reaction of this diradical with the solvent or to absorption in a different spectral region.

Experiments at low temperatures in rigid media should be more successful in detecting this species.

CONCLUSIONS

The close correlation between the results obtained above and those for simple quinones is evidence that very similar species are formed in both cases and that the reactions taking place on light absorption, as well as the behaviour of the radicals under various conditions, are similar. However, it should be stressed that the solutions studied above in all probability contained alcohol-soluble impurities from the commercial dyestuff which may influence the photochemical behaviour of the pure dye, although the great similarity to the radicals produced by the rigorously purified dyes referred to above [21] seems to indicate that they have only a minor effect. It is not proposed to comment in detail here on the relationship between the structure of the quinone, its excited states, and its activity, though it is hoped to present such a correlation in a forthcoming paper. However, if reference is made to Venkataraman [2] or to one of the authors already referred to e.g. [1, 4, 23], it will be observed that all the dyes quoted here contain essentially anthraquinone, substituted to a greater or lesser extent. The production of semiquinone radicals and radical-ions and the close similarity in behaviour of all these dyes must owe their explanation to this fact, and are considered to be a justification for using the above methods to help understand the practical case of vat dyes on fibres.

BRITISH RAYON RESEARCH ASSOCIATION
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(*Received 9th September 1958*)

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Durable Pleating of Wool Fabrics

We are indebted to the Commonwealth Scientific and Industrial Research Organisation of Australia for the development of the commercial process for the durable creasing and pleating of wool fabrics [1]. The process associating the trade-mark *Si-ro-set* with the approved reagent solution entails treatment of the wool fabrics with a dilute solution of ammonium thioglycollate and pleating in a steam press or autoclave. The properties of ammonium thioglycollate are essentially those of a reducing agent, but it is also capable of reacting with metals. Both functions are clearly demonstrated when the effect of the treatment is examined on dye ranges. For the fastness details reference should be made to the recent ICI publication [2].

As might be expected, those dyes which are known to be highly sensitive to reduction are among those most severely affected. Coomassie Navy Blue 2RNS (C.I. Acid Blue 113) provides an example of this type, and under more severe conditions of treatment in an autoclave it is reduced to an orange. The effect on metal-containing dyes is, however, less clear, for although few of the chrome range are completely unaffected, many are satisfactory and only a few are very poor. The yellows are particularly poor as a group. In general, however, the effect on the dyes seems to be specific for individual members and not characteristic of a class or range. An exception to this is provided by the premetallised dyes of 1:1 chromium-complex type, such as the Ultralans (ICI) and the Neolans (Ciba), which undergo an appreciable colour change under treatment, and this behaviour dominates the range as a whole. Whilst with all other dyes examined the effect of the treatment is irreversible, the Ultralan types provide a further exception in that the original colour can, with most dyes, be almost completely restored by a mild oxidation treatment, say with 2-vol. hydrogen peroxide. Even though the means of colour restoration is at our disposal, we cannot expect to make this range any more attractive to the trade, as the double processing which it necessitates (the second process being oxidation) is bound to meet with stiff resistance. Nevertheless, the observation can be of some value as a means of colour restoration whenever goods dyed with these dyes are inadvertently subjected to the Si-ro-set treatment.

Dye	*Colour Index* Designation	Si-ro-set (Hoffman press)	Oxidation (2-vol. H_2O_2)
Ultralan Yellow GS	C.I. Acid Yellow 54	2 Redder	4–5 Redder
Ultralan Orange GS	C.I. Acid Orange 74	2 Redder	4–5 Redder
Ultralan Orange GRS	C.I. Acid Orange 62	2 Redder	4 Redder
Ultralan Red BS	C.I. Acid Red 212	2 Bluer	4–5 Bluer
Ultralan Bordeaux RS	C.I. Acid Red 179	2 Bluer	4 Bluer
Ultralan Bordeaux BS	C.I. Acid Red 187	1 Bluer	4 Bluer

The above selection of some examples from the Ultralan range will demonstrate the extent of recovery of colour. The assessments were carried out, with the S.D.C. Grey Scale for assessing Change in Colour, on dyeings at 1/1 ISO standard depth.

G. M. SWINTON

DYEHOUSE DEPARTMENT
IMPERIAL CHEMICAL INDUSTRIES LTD.
DYESTUFFS DIVISION
HEXAGON HOUSE
MANCHESTER 9

7th January 1959

[1] C.S.I.R.O., *Report* No. G6 and *Trade Circular* No. 4.
[2] ICI, *Technical Information* (*Dyehouse*) No. 459.

Notes

Meetings of Council and Committees

February

Council— 4th

Publications— 17th

International Federation 1959 Congress—

Planning and Co-ordinating 2nd

Social Subcommittee 6th

Ladies' Subcommittee 6th

Review of Textile Progress— 16th.

Death

We regret to report the death of Mr. D. E. Doran.

Tentative Definitions

The Terms and Definitions Committee would welcome comments on the following tentative definitions. Such comments should be addressed *in writing* to the Editor and Technical Officer, Society of Dyers and Colourists, 19 Piccadilly, BRADFORD 1, Yorkshire. The Committee also invites requests for definitions of terms which may have given rise to special difficulty.

accelerant

A substance, often a swelling agent, which, when added to a dyebath, accelerates the diffusion of dyes into a fibre.

acid dye
(revised definition)

An anionic dye (q.v.) so called because many of the class are characterised by their substantivity for protein fibres when applied from an acid dyebath.

beck

See drawing of *winch dyeing machine.*

carrier

A term used to describe a type of accelerant, particularly when used in the dyeing of hydrophobic fibres with disperse dyes.

cover roller

A roller engraved with a delicate pattern, e.g. vertical or diagonal lines, scrolls, spots, checks, etc. It is used for overprinting an existing print to obtain a distinct though subdued patterned ground. The operation is termed *covering.*

covering

See *cover roller.*

finish

(*a*) The final result of all the processes applied to the material to convert it into a desired state.

Note— This may or may not include coloration. The noun may be made more specific by reference to special effects such as *mercerised, water-repellent, crease-resistant,* etc.

(*b*) The substance (other than a colorant) which has been added to the material to produce a desired state.

mangle

Any machine whose purpose is to express liquid from moving textiles by passage through a nip. The textile may be in rope form or in open width, and the mangle may consist of two or more rollers (bowls) running in contact.

multibox

See *open soaper.*

Note— Use of this term is deprecated.

nip

See *bowl.*

nip padding

The even impregnation of a textile material in open width by passage through a padding mangle (q.v.) wherein the padding liquor is carried as a film on one of the bowls comprising the nip and is transferred to the dry material as it passes to the nip.

open soaper

A machine for continuous wet treatment of textiles in open width, consisting essentially of a number of compartments, each with several top and bottom rollers which the material passes over and under in its passage through the liquor. Frequently the material goes through a heavy nip in its passage from compartment to compartment in order to limit the carry-through of liquid. By suitable arrangement of the liquids in the series of compartments a sequence of operations such as fixing, rinsing, soaping, and rinsing can be carried out effectively and economically. Individual machines show variation in methods of heating, agitating, and circulating the liquor and in the means by which the textile is drawn through mechanically.

optical bleaching agent
optical brightening agent

See *fluorescent brightening agent.*

Note— Use of these terms is deprecated.

pad

See *padding mangle.*

padding
(revised definition)

The even impregnation of a textile material in open width by means of a padding mangle (q.v.).

padding mangle

A form of mangle for even impregnation of textiles in open width in which the textile is passed through one or more nips. The textile may be saturated before passing through the nip, as in *slop padding*; or the impregnating liquid may be carried as a film on the surface of one of the bowls comprising the nip and transferred to the textile as it passes through the nip, as in *nip padding*.

peg rail

See drawing of *winch dyeing machine*.

print pad

An intaglio roller used in printing for applying thickened dye or chemical to the whole area of the textile, as distinct from local application of a design, for such purposes as surface mercerising or treatment with anti-scumming compounds.

Note— Back-filling, suction-hydroextracting, etc. are excluded, although they may be known locally as "padding".

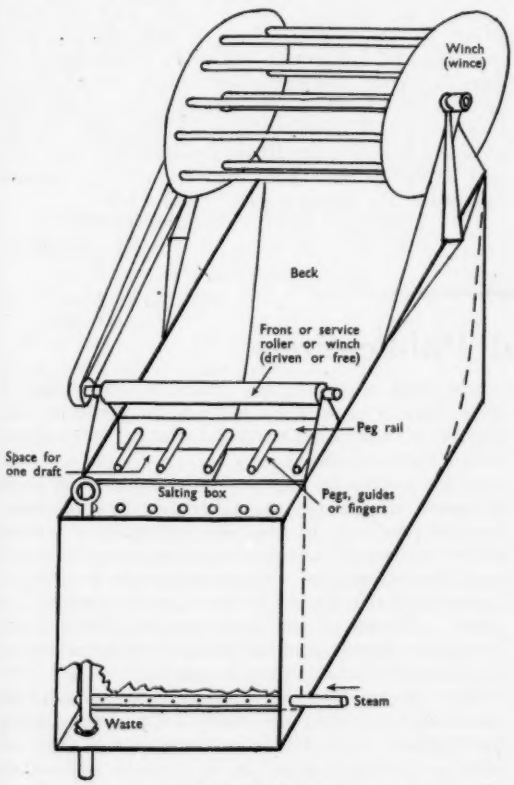
salting box

See drawing of *winch dyeing machine*.

setting
(revised definition)

The process of conferring stability on fibres, yarns, or fabrics, generally by means of moist or dry heat.

Note— Extensive notes amplifying this Textile Institute definition in the cases of synthetic-polymer materials and of natural fibres and rayons will be found in *Textile Terms and Definitions* (Manchester: The Textile Institute. 3rd edition 1957), pp. 88–89.



Winch Dyeing Machine

slop padding
(revised definition)

The even impregnation of a textile material in open width by passage through a padding mangle (q.v.) wherein the material is saturated with the padding liquor before reaching the nip.

swelling agent

A substance which causes the total liquid imbibition of a fibre to increase.

Note— A swelling agent may be used in a dyebath or a print paste to promote coloration by accelerating the diffusion of dyes into a fibre.

tailing

A gradual and progressive change in colour along a length of material to which colorant has been applied by padding or other continuous technique.

wince

Local variant of *winch* (q.v.).

winch

A horizontal rotor, commonly circular or elliptical in cross-section, used for lifting cloth, in rope form or in open width, during processing (see drawing).

winch dyeing machine

A dyeing machine consisting essentially of a dye vessel over which a winch rotates and moves a length of cloth joined end to end through the liquor (see drawing).

Centenary of the Discovery of Fuchsine (C.I. Basic Violet 14)

The centenary was celebrated at Lyons on 18th October 1958, the programme having been arranged on the initiative of the Union Syndicale des Fabricants de Matières Colorantes et d'Hydrosulfites in association with the Union des Industries Chimiques, the Groupement Interprofessionel Lyonnais, the Fédération de la Soierie, the Union des Syndicats de la Teinture, de l'Impression, de l'Apprêt de Lyon et de la Région, the Groupement des Syndicats Patronaux des Industries Chimiques et Connexes de la Région Lyonnaise, and the Association des Chimistes de l'Industrie Textile. The celebrations were held under the patronage of the Minister of Industry and Commerce, the Prefect of the Rhône, the Mayor of Lyons, the Rector of the University of Lyons, and the President of the Chamber of Commerce of Lyons. The functions formed part of the bimillennary celebrations of the town of Lyons.

Fuchsine was first made in 1858 by François-Emmanuel Verguin, born at Lyons in 1814, and among the 300 or so persons who attended the centenary celebration were several of his grandchildren and great-grandchildren. In the morning, at the Palais de la Bourse, M. Ernest Sack gave a biographical sketch of Verguin and discussed the significance of his discovery (this lecture is printed in *Teintex*, **23**, 849–864 (Dec. 1958)).

At a civic reception at the Hôtel de Ville it was mentioned that a street in Lyons is named after Verguin. After lunch in the Chalet du Parc a short pilgrimage was made to Roussillon, where a wreath was placed on Verguin's grave. There followed a visit to the "Maison Verguin", where the inventor spent the latter years of his life and which is now maintained as a hostel for technologists at the Péage de Roussillon works of the Société Rhône-Poulenc. A small exhibition was arranged at the

Chalet du Parc. The day concluded with a reception and a visit to the works of the Rhône-Poulenc concern.

Jubilee of Continental Associations of Colourists

The Schweizerischer Verein der Chemiker-Coloristen celebrated this jubilee on 29th November 1958 in Zürich. In the morning, in the chemistry lecture theatre of the Federal Polytechnic, lectures were given by Dr. H. Zollinger and Dr. F. Hügli on *Molecular Structure and Dyeing Processes* and *The Physicochemical Bases of Dyeing* respectively—these papers are to be published later.

Lunch in the Hotel Elite was attended by about 170 persons. Dr. A. Locher conveyed greetings from the Basle dyemakers, and discussed the development of the Swiss textile industry. There were hopes that a chair of textile chemistry might soon be founded in the organic technology department of the Federal Polytechnic in Zürich, and developments were also in progress at the "Technikum" level.

Dr. H. Ris outlined the history of the associations since the first announcement in December 1908 (cf. J.S.D.C., **74**, 39 (1958)), and stressed the part played by the Swiss in maintaining international relations between colourists. Finally Herr A. Bodmer gave his reminiscences of the early days in the textile-printing town of Königinhof in Bohemia, and drew a comparison with conditions in Herisau in Switzerland, where, too, brewers and dyers tended to congregate together.

OBITUARY NOTICE

Frank Leslie Barrett

Frank Leslie Barrett, Fellow of the Society, died in November 1958, shortly after his retirement from the post of Director of Research of the Bleachers' Association Ltd. He was born in Macclesfield in 1893 into a family which had had long connections with the silk industry. His father became Lecturer on Textile Design at Bradford Technical College, at which Barrett later gained his Diploma in Chemistry and Dyeing. He gained a Royal Scholarship to the Royal College of Science, but was able to stay only for one year owing to the outbreak of the First World War. After a short time with the Gas Corps in France, he, with other chemists, was recalled to do research at the Royal Arsenal, Woolwich, where he joined a team working under W. H. Gibson on the purification of cellulose for "nitro-cotton" and its control by viscosity methods.

Shortly after the end of hostilities Barrett joined the new Research Department of the Tootal Broadhurst Lee Co. Ltd. In 1925 he joined the Bleachers' Association Ltd., becoming manager of their experimental works at Turton. On the closure of these in 1930, he went to the firm's laboratories in Manchester with Wilfred Kershaw, and was later made manager of the Association's dyeworks at Leyland, where he remained until the end of the Second World War. He was appointed the first Director of Research at the new Bromley Cross laboratories in 1947. In the course of his career Barrett carried out many technical missions to the Continent and the U.S.A. He was responsible for many patents on bleaching, dyeing, and finishing.

Barrett was a very practical man: he was at home with machinery of all kinds, and was very adept in the construction of instruments and experimental apparatus. He was a genial fellow in work and play, and he carried a liberal share of the artistic streak which ran through the family (his brother, A. C. Barrett, was a well known cartoonist). He was a Fellow of the Royal Institute of Chemistry and a Fellow and Life Member of the Textile Institute. He leaves a widow and a son.

F. C. Wood
A. E. Stubbs

New Books and Publications

Chemie der Azofarbstoffe

By Heinrich Zollinger. Pp. 308. Basle: Birkhäuser Verlag. 1958. Price, Swiss francs 36.25 (DM 36.25).

The author, Dr. Zollinger, whose work on reaction kinetics is well known, should need no introduction to those engaged in the dyemaking industry or to students of dye chemistry, to whom this book makes especial appeal.

The previous works of reference in this field have been those of— Hantzsch (*Die Diazo Verbindungen*), Cain (*The Chemistry and Technology of the Diazo Compounds*), Saunders (*The Aromatic Diazo Compounds*), and Holzacher (*Die aromatische Diazo Verbindungen*), the latter two publications having appeared in 1947.

This newer publication is a very worthy successor, in that it covers the chemistry of the whole field of azo dyes. Much of the material given here is based upon lectures delivered by the author at the University of Basle, with more recent material added.

In his preface Dr. Zollinger states that there is no intention of giving comprehensive lists of commercial products, but his aim is to give a survey of the whole field, which he does in a clear, concise, and attractive form. Those fortunate enough to have heard Dr. Zollinger or to have studied his many publications will appreciate this book from his hands, knowing of his methods of approach to the chemistry of azo dyes in general.

The preparative and technological aspects of azo work have been previously dealt with in many publications, but this book is unique in that no similar coverage with as up-to-date references (e.g. to Procions and Remalans) has hitherto been

incorporated in book form. The book in itself is extremely well produced and is divided into fifteen chapters, the first six dealing with the diazo compounds. Methods of diazotisation, equilibria, isomerisation, and decomposition reactions are surveyed and the kinetics explored very thoroughly, the whole being clearly referenced. Similarly treated are the next two chapters, dealing with the diazoamino and the azo compounds. The author has drawn very widely from his own researches into the mechanism of the coupling reaction and its application to the technology of azo dyes in chapters 9 and 10.

A technical review is given in chapter 11, and at least one example of each type of azo dye is given. One could have wished that chapter 12 had been expanded to show the relationship of the structure, with regard to the commonly used groups in the molecule, to absorption maxima on the one hand and to selected properties on the other. However, perhaps the author believes that the only way really to appreciate research is to consult the original papers, and these are referenced.

The following chapters deal with the chemistry of the formation of the metal-complex dyes and finally a study of the application of azo dyes to protein, cellulosic, and synthetic fibres, together with a survey of the physicochemical basis of the dyeing process.

Experimental details are, of course, lacking in a book of this type, but the numerous references to published information are a welcome extension to the text, which in itself is very well presented.

The book has author and subject indexes, the former containing over 900 references, which is evidence of the work which has gone into the preparation of this book. It certainly fills a gap in technical literature, and should be in the hands of every serious student of dye chemistry.

A. GOTHARD

Fundamentals of High Polymers

By O. A. Battista. Pp. ix + 140. New York: Reinhold Publishing Corpn. (London: Chapman & Hall Ltd.) 1958. Price, $5.50 (44s. 0d.).

Most textbooks on high-polymer science have been primarily designed for the graduate chemist or physicist, and there are few texts suitable for the student. This book, however, has been designed to serve as an introductory textbook on high polymers at the undergraduate level. The first part deals with basic principles of high polymers. Fundamental terms and definitions are clearly and precisely defined in the first chapter, and a subsequent one outlines the more important aspects of the chemistry of natural and related polymers, addition and condensation polymers, stereospecific polymers, network polymers, and inorganic polymers. The second part is concerned with the formation of products from polymer chains. The architecture and the geometry of polymers and the factors determining crystallinity are first considered, and methods of determining chain length and chain-length distribution are briefly discussed. A chapter on the solid state of polymers deals with orientation, crystallite size, and polyphase structure. Another deals briefly with physical properties, including tensile strength, flow, swelling, compatibility with dyes, and moisture sensitivity, and outlines some measurable properties. A final very short chapter makes some suggestions as to the future of high polymers.

To some extent the book fulfils the aim of providing an undergraduate introductory text. However, the treatment of certain topics is very brief. Thus, the important topic of polymerisation is very briefly discussed, and number-average and weight-average molecular weights are not defined mathematically. Aspects of fibre-forming polymers are, perhaps, overstressed. Errors and omissions seem to be few. However, membranes for osmometry do not usually consist of porous glass or metal discs as stated in the text, and the term "dynamic osmometer" is not always used in the sense given on p. 82. Infrared absorption studies of polymers and glass temperatures are not mentioned.

Such omissions and the perhaps rather superficial treatment of some topics are mitigated by an exhaustive bibliography of text and reference books covering the main aspects of polymer science. The book is very readable, and some clear diagrams help to clarify certain concepts. It should prove useful to all requiring an outline of the main fundamentals of polymer science and as a basis for the understanding of more advanced texts. Within the limits mentioned, students will find it a useful introductory text. It should, however, be emphasised that those preparing for examinations in high polymers at degree or equivalent level, such as the A.S.D.C. examination, will require considerable additional material either from lectures or from more advanced textbooks.

W. R. MOORE

Physical Chemistry of High Polymers

By Maurice L. Huggins. Pp. xiii + 175. New York: John Wiley & Sons Inc. (London: Chapman & Hall Ltd.) 1958. Price, 52s. 0d.

Dr. Huggins has played an important part in the development of polymer science and has made significant contributions to many of its branches. This book is based on notes used by him while a visiting lecturer in Japan. In sixteen concise chapters he deals successively with nomenclature and classification; synthesis; variations in composition and structure; chain configurations; simplified and revised thermodynamic theories of polymer solutions; phase equilibria; viscosities of dilute solutions; plastic flow; long-range elasticity; typical addition and condensation polymers; the structures of some carbohydrate polymers; the structures of synthetic polypeptides, keratin, and silk; the structure of collagen; and the structure of corpuscular proteins.

As he states in the preface, his own contributions are emphasised, and no attempt is made to deal comprehensively with the work of others. As a result, many topics and concepts that one would expect to find discussed in some detail in a book apparently surveying the physical chemistry of

polymers are omitted or receive very brief treatment. For example, addition and condensation polymerisation are covered in a chapter of only nine pages. Experimental methods are not considered in any detail. Second-order transitions, polymer degradation, and molecular-weight distributions are accorded only a paragraph each. The treatment of topics considered in more detail inevitably suffers from the author's emphasis on his own contributions, resulting in the omission of other concepts and opinions in many cases. This is, perhaps, most evident in the chapter dealing with long-range elasticity, which is largely discussed in terms of a model treated by Dr. Huggins in 1946. The book can hardly be regarded as providing a comprehensive survey of the physical chemistry of high polymers.

In spite of these defects, there is much in the book to interest and stimulate the discriminating polymer chemist. The chapter on revised thermodynamic theories of polymer solutions contains an interesting and hitherto unpublished treatment of heat and entropy changes occurring on mixing polymer and solvent. The later chapters describing the molecular structures of synthetic and natural high polymers (including proteins) will be of considerable interest to many readers of this *Journal*. The text is generally concise and there are many clear diagrams. In view of the author's preoccupation with his own work and the consequent uneven treatment, the book is not generally suitable as a text for student use.

W. R. MOORE

Epoxy Resins

By Irving Skeist, assisted by George R. Somerville. Pp. xiii + 293. New York: Reinhold Publishing Corpn. (London: Chapman & Hall Ltd.) 1958. Price, 44s. 0d.

This book is the eighth of a series of monographs published as the *Reinhold Plastics Applications Series*, and as this implies, the treatment is primarily technological. Only sufficient of the theoretical chemical background is given to make the technological processes understandable. The book is not claimed to be for chemists but is intended for design engineers, the large body of users of plastics, and students in technical colleges. In view of this declared aim, the reviewer can see little value in introducing a special symbolism for the epoxy resins which is used only on pp. 13, 14, 22, and 23, and could be safely used only by a trained chemist. Furthermore, the treatment of di-, tri-, and tetra-functional molecules on p. 15 and the diagrams on the next page would not satisfy the chemist, and would be misleading to the non-chemist. However, these are relatively minor points.

After an introductory chapter, two chapters follow on the chemistry of the resin intermediates and the curing of liquid epoxy resins respectively. The latter chapter, apart from the summary chemical treatment, deals with the various curing agents in a rather encyclopedic way, but useful for reference purposes. It might be noted here that many of these resin intermediates and curing agents are proprietary products available in the U.S.A., and this could limit the uses of the book in this country to some extent.

The remaining and greater part of the book is purely technological, covering such aspects as modifications to the resins—"flexibilising", introduction of reinforcements, fillers, thixotropic agents, etc.—and applications—tooling for metal working, glass-reinforced "epoxies" for structural uses, particularly for pipes in the petroleum and chemical industries, embedding electrical components, adhesives—and throughout this section there are representative formulations based on proprietary products. A long chapter, written by George R. Somerville, deals with the properties and applications of epoxy resins in surface coating, again with formulations, for various types of varnish, mastic, etc.

The bibliography contains just under 300 references to articles and papers of technological importance.

W. S. SHORT

New Books received

Nouvelles Tables de la Colorimetrie appliquée à la Physiologie de la Couleur. FRANTZ BRAUN. Tome **I**, Pp. 80 + 2 colour plates. Tome **II**, Pp. 124. Tome **III**, Pp. 136. Brussels: Edition–Librairie–Publicité Internationales. 1957. Belgian francs 1,500.

Veredler-Jahrbuch— Deutscher Färberkalender für das Jahr 1959. **63** Jahrgang. O. MECHEELS (editor). Munich: Franz Eder Verlag. Pp. iv + 64 + vi + 432 + 64 + 64. [1958]. DM 13.50 ($3.50).

The Chemistry and Technology of Leather. Volume **II**. *Types of Tannages.* F. O'FLAHERTY, W. T. RODDY, and R. M. LOLLAR (editors). New York: Reinhold Publishing Corpn. (London: Chapman & Hall Ltd.) 1958. Pp. xiv + 554. 132s. 0d.

Vorbehandlung und Färben von synthetischen Faserstoffen. H. U. Schmidlin. Basle: Verlag Schweizerische Vereinigung von Färbereifachleuten. 1958. Pp. viii + 358. Swiss francs 20.00.

Epoxy Resins. IRVING SKEIST, assisted by GEORGE R. SOMERVILLE. New York: Reinhold Publishing Corpn. (London: Chapman & Hall Ltd.) 1958. Pp. xiii + 293. 44s. 0d.

Reports on the Progress of Applied Chemistry. Volume **XLII**. 1957. H. S. ROOKE (editor). London: Society of Chemical Industry. [1958]. Pp. 910. 100s. 0d.

Review of Textile Progress. Volume **9**. 1957. Manchester and Bradford: Textile Institute and Society of Dyers and Colourists. 1958. Pp. 545. 42s. 0d. ($6.00).

Publications and Titles of Theses 1955–1956. Leeds: The University. 1958. Pp. 74. 6d.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index*

I— PLANT; MACHINERY; BUILDINGS

Power and Heat Supply for Textile-finishing Plant

R. Quack

Z. ges. Textilind., **60**, 707–714 (25 Aug. 1958)

A review covering— (i) fuel and heating, including some experiences with oil fuel, (ii) feed water and steam generation, (iii) heat and power requirements, (iv) measurement and control. J.C.F.

Three-phase Multi-motor Drives in Textile Finishing

G. Schindler

Melliand Textilber., **39**, 1272–1275 (Nov. 1958)

New developments in speed controls for three-phase A.C. commutator motors are illustrated in applications to textile finishing machinery where less economical D.C. drives were previously employed. S.M.J.

Corrosion-resistant Steels for Bleaching and Dyeing

L. Wetternik

Melliand Textilber., **39**, 1152–1156 (Oct. 1958)

The author reviews reductive activation and oxidative passivation of metals, the raising of the potential of iron by chromium, and the effect of carbon content on the critical chromium concn. required on account of carbide formation. Corrosion rates of chromium and chromium–nickel steels in sulphuric and hydrochloric acids and the effect of alloying are graphically illustrated. Intergranular and pitting corrosion and their prevention are depicted. The composition of 9 Austrian corrosion-resistant steels is tabulated. Ten graphs summarise their corrosion behaviour and its variation with temp. and concn. of HCl and H_2SO_4. Very high stability is exhibited by Antinit SAS 10 (18/22/3/2 Cr, Ni, Mo, Cu + Nb 8 × C; C ⩾ 0·07%) towards H_2SO_4 and by Super Antinit C (16/60/17 Cr, Ni, Mo; C ⩾ 0·10%) towards HCl. S.M.J.

Corrosion of Apparatus by Food Dyes

A. V. Avdeera, A. L. Sokolovskiĭ and P. A. Tsganora

Khlebopekar. i Konditer. Prom., **2**, (7), 14–16 (1958): *Chem. Abs.*, **52**, 20759 (25 Nov. 1958)

Comparison of the effect of food dyes and essences on Cu, duralumin, chrome nickel steel, chrome steel, Al and plastic coatings of vinyl chloride, polyethylene and poly-(trifluorochloroethylene) at 70°c. lead to the recommendation that plant used for producing food colours and essences should be made of chromenickel or chrome steel or be coated with poly(trifluorochloroethylene). C.O.C.

Rayon Hank Dyeing

L. L. Walmsley

Modern Textiles, **39**, 40 and 42 (March 1958)

Poor winding characteristics after dyeing fine deniers and speckiness in vat dyeing of heavier deniers are common faults in rayon hank dyeing. The Cascade machine, claimed to be the most suitable machine for hank dyeing, carries the hanks on horizontal tubes, perforated on top, through which dye liquor is pumped. Horizontal arms rotate the hanks at frequent intervals. Better dyeing of heavier deniers results when plastic covers are fitted closely over the hanks, and better winding characteristics in fine deniers result with open mesh covers fitted over the hanks. Recommended procedure is given for hank dyeing with vat and direct dyes in this machine. E.C.

Continuous Card Wire Raising Machine

J. Ernestus

Melliand Textilber., **39**, 1128–1132 (Oct. 1958)

A non-stop machine is described, illustrated, and shown to be faster than conventional types. S.M.J.

Rotary Press as Final Finishing Machine

H. Herkenrath

Melliand Textilber., **39**, 1132–1134 (Oct. 1958)

The factors influencing application in practice are considered. S.M.J.

PATENTS

Apparatus for Reheating the Oil Feed to a Carbon Black Furnace

Phillips Petroleum Co. *USP* 2,844,443

Drying Wet Carbon Black (C.I. Pigment Black 6 and 7) Pellets

Phillips Petroleum Co. *USP* 2,843,942

Apparatus wherein wet pellets are dried and the water and Carbon Black dust obtained are recovered and recycled to a wet pelleting operation C.O.C.

Drum Sieve Dryers for Fibre Fleeces

Fleissner & Sohn Maschinenfabrik *BP* 806,287

A machine in which as the fleece becomes drier its thickness on the drum sieve is increased thus enabling machines of much shorter length to be used. C.O.C.

Wet Processing of Granular Material, Loose Fibres or Yarn Packages

Aktiebolaget Pluria *BP* 806,883

A package dyeing machine which can also be used as a pressure dyeing machine has a valve system which enables the direction of pressure in the dye liquor to be rapidly varied, the resulting pulsations in the liquor causing rapid exhaustion of dye into the fibre. C.O.C.

Transport of Pieces through Chambers or the like

Aktiebolaget Svenska Fläktfabriken *BP* 806,332

The goods are carried on an endless band between fixed loading and unloading points, the band moving intermittently and being carefully synchronised with other parts of the manufacturing process. C.O.C.

Controls of Moving Fabric, Paper, etc.

ICI *BP* 806,598

Apparatus for preventing lateral deviation in fabric, etc., as it passes over or on to rollers. C.O.C.

Pressing Garments on Formers

S. G. Widdowson *BP* 807,105

Machine for Applying Transfers to Hosiery and Other Garments

Paramount Textile Machinery Co. *USP* 2,843,956

1958 Reviewed— Technical Developments in the Paint and Printing Ink Industries (V p. 169)
Shrinking and Crimping Fabric (X p. 176)

III— CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Magnetochemical Study of Cyanuric Acid, Melamine, and some Derivatives

Y. Matsunaga and S. Morita

Bull. Chem. Soc. Japan, **31**, 644–647 (July 1958)

Measurements of the magnetic susceptibilities of melamine, cyanuric acid, and eight of their derivatives, and a discussion. A.J.

Periodate- and Hypochlorite-oxidised Starches— Viscosity Behaviour

R. L. Mellies, C. L. Mehltretter, and I. A. Wolff

Ind. Eng. Chem., **50**, 1311–1314 (Sept. 1958 Pt. I)

Hypochlorite-oxidised starches are now used in preference to native starches in paper sizing because of their excellent adhesive powers, higher fluidity, and lower gelatinisation temperatures. Various oxidised starches, prepared from the same corn starch by treatment with sodium hypochlorite (type I) and sodium metaperiodate (type II), were compared, particularly for viscosity behaviour. The more highly oxidised members of each type gelatinised and broke down rapidly. In the presence of NaCl the viscosities of type II were not reduced to the same extent as those of type I. De-ashing lowered the viscosities of type I appreciably. The viscosities of Type II were decreased by higher temp. and longer times of drying during preparation and by storage. W.K.R.

Gum Ghatti (Indian Gum). III— Neutral Oligosaccharides formed on Partial Acid Hydrolysis of the Gum

G. O. Aspinall, B. J. Auret, and E. L. Hirst

J.C.S., 4408–4414 (Dec. 1958)

Mothproofs containing Acid Amide Groups
H. Martin
Textil-Rund., **13**, 439–457 (Aug.); 571–585 (Oct. 1958)

The characteristics of Eulan CN (FBy) and Mitin FF (Gy) are considered in the light of their constitutions. Consideration of other compounds claimed from time to time shows that, as regards toxicity towards moth-grubs and dermestid beetles, monosulphonic acids are more effective than disulphonic acids, a central position of the sulpho group, e.g. *ortho*, in the molecule is better than an end position, the nucleus carrying the sulpho group is better if halogenated, and a trifluoromethyl group is preferable to halogen alone. Acid amide aromatic sulphonic acids and ammonium compounds show great biological activity; carboxylic acid amide groups in conjunction with phosphonium compounds are less effective. Mitin N (Gy) is an aryl carbamic acid amide carrying trifluoromethyl groups and no solubilising groups.
S.R.C.

PATENTS

Textile Lubricant
Esso Research & Engineering Co. *USP* 2,839,464

A textile lubricant readily removable by scouring with soap and soda consists of (1) a mineral oil containing no constituents which ionise in aqueous solution to yield ions forming compounds of low water solubility, (2) a little of a detergent of formula $R^1{\cdot}CO{\cdot}O(C_nH_{2n}O)_x{\cdot}H$ (R^1 = unsatd. aliphatic radical of 14–22 C; n = 2–6; x = 5–30), e.g. Antarox Agent 377–AE (G), (3) a little of an emulsifying agent of formula $R^2{\cdot}(OC_mH_{2m})_y{\cdot}OH$ (R^2 = Ar, aralkyl or sat. or unsat. Alk of 10–20 C; m = 2–6; y = 2–20), e.g. Triton X-45 (Rohm & Haas Co.), and (4) as a coupling or stabilising agent an oil-soluble unsaturated fatty acid partial ester of a higher polyhydric alcohol of the type disclosed in *USP* 2,404,240, e.g. sorbitan monooleate.
C.O.C.

Antistatic Composition
Union Carbide Corpn. *USP* 2,841,567

A durable antistatic finish is obtained on hydrophobic polymers by use of a 4–12% solution of an ester of formula $CH_2{:}CHCOO(CH_2CH_2O)_nR$ (R = H or acrylyl; n = 7–22) containing 1–16% (on dry weight of the ester) of borax, boric acid or an inorganic borate, and 5–25% (on dry weight of the ester) of peracetic acid and/or H_2O_2.
C.O.C.

Size for Polyester Yarns
Monsanto *BP* 807,185

Water-soluble salts of interpolymers of $\leqslant$ 25 mol.% of a conjugated 1:3-diene, e.g. butadiene, and $\geqslant$ 75% acrylic or methacrylic acid are good sizes for polyester yarns.
C.O.C.

Rendering Polyvinyl Halide Resins Adherent to Glass
General Aniline *BP* 806,936

Addition of 2–3% of a polymer of *N*-vinylpyrrolidone to polyvinylhalide resins makes them highly adherent to glass.
C.O.C.

Water-repellent Agents and other Products obtained from Carbohydrates
General Aniline *BP* 806,935

Carbohydrates when dissolved in 2-pyrrolidone or *N*-methyl-2-pyrrolidone react readily with various reagents to give a variety of compounds having wide fields of use. Thus by treatment with anhydrides ether-soluble products are obtained, treatment with phosphorus tri- or pentachloride (or bromide or iodide) results in one or more hydroxyl groups of the carbohydrate being replaced by halogen. In one example sucrose dissolved in *N*-methyl-2-pyrrolidone was heated with stearyl chloride to yield the waxy octastearyl sucrose. A tetrahydrofuran solution of this product renders cotton water-repellent.
C.O.C.

Fungicides
Hercules Powder Co. *USP* 2,844,506

Lower alkyl-substituted (preferably in the *para* position) benzoic acids and their Cu and Zn salts have strong fungicidal properties. Thus bleached cotton impregnated with 1% of zinc cumate or copper *p-tert.*-butyl benzoate lost little strength after a 14 days soil burial test in which the untreated control was completely destroyed. C.O.C.

Organophilic Hydrophobic Silicates, Silicas, Silicic Acid, and Hydrated Silicas—Fillers for Rubber and Plastics and Vulcanisers for Silicone Rubber (IV p. 167)

1958 Reviewed—Technical Developments in the Paint and Printing Ink Industries (V p. 169)

Carboxymethylcellulose Acids and Complex Ammonium Zirconium Carbonates for forming Water- and Alkali-resistant Coatings (V p. 170)

Bleaching Composition (VII p. 172)

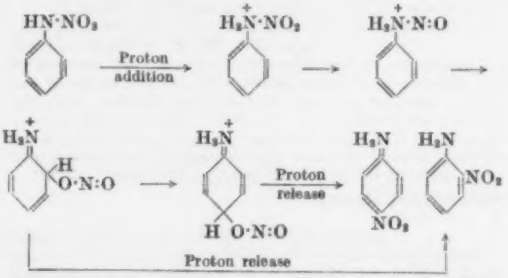
Extenders for Oil-in-Water Emulsion-type Printing Pastes (IX p. 175)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Nitration at Nitrogen and Oxygen Centres. I—Kinetics and Mechanism of the Conversion of Secondary Amines into Nitroamines
E. D. Hughes, C. Ingold, and R. B. Pearson
J.C.S., 4357–4365 (Dec. 1958)

Rearrangement of Aromatic *N*-Nitroamines. II—Isotopic Test for Intramolecular Character in the Nitro Transfers from Side-chain to *ortho* and *para* Positions
S. Brownstein, C. A. Bunton, and E. D. Hughes
J.C.S., 4354–4357 (Dec. 1958)

Previous work indicated that the main part of the rearrangement of *N*-nitroaniline in aq. H_2SO_4, which produces 93% of *o*- and 7% of *p*-nitroaniline, is intramolecular, but it was not quant. enough to exclude the possibility that the *para* product and perhaps a little of the *ortho* isomer might arise from a concurrent acidolysis of the *N*-nitro group and subsequent *C*-nitration by the nitric acid formed. This possibility is now excluded by showing that no trace of a nitrogen-isotopic label contained in nitric acid introduced into the medium used for the rearrangement appears in either the *o*- or the *p*-nitroaniline. Both products are therefore formed exclusively by intramolecular mechanisms, for which the following scheme is suggested—



H.H.H.

Oxidation of Aromatic Amines. VII—Action of Persulphate on some Aromatic Amines
E. Boyland and P. Sims
J.C.S., 4198–4199 (Nov. 1958)

Previous work on persulphate oxidation has been extended to include the secondary amines, *N*-methylaniline and diphenylamine and the heterocyclic amines 2- and 4-aminopyridine, all of which yield the corresponding *ortho*-subst. sulphuric esters; 4-aminopyridine yields also 4:4′-azoxypyridine. 4-Amino-2′:3′-dimethyldiphenyl affords 4:4′-azo- and 4:4′-hydrazo-2′:3′-dimethyldiphenyl as well as the expected sulphuric ester. 3:6:1- and 2:6:1-Xylidine afford sulphuric esters; in the latter amine, where both positions *ortho* to the amino group are blocked by methyl groups, substitution by the entering sulphuric group is in the *para* position. H.H.H.

(3-Substituted 2-Naphthyloxy)-n-alkanecarboxylic Acids
D. Woodcock and B. L. Davies
J.C.S., 4723–4728 (Dec. 1958)

The preparation and properties are described of 3-substituted 2-naphthyloxyacetic acids (with 3-substituents F, Br, I, CH_3, OH, $O{\cdot}CH_3$, NO_2, CN, $CO{\cdot}NH_2$, COOH). The preparations of 3-fluoro-2-naphthol and 3-methyl-2-naphthol are also included. H.H.H.

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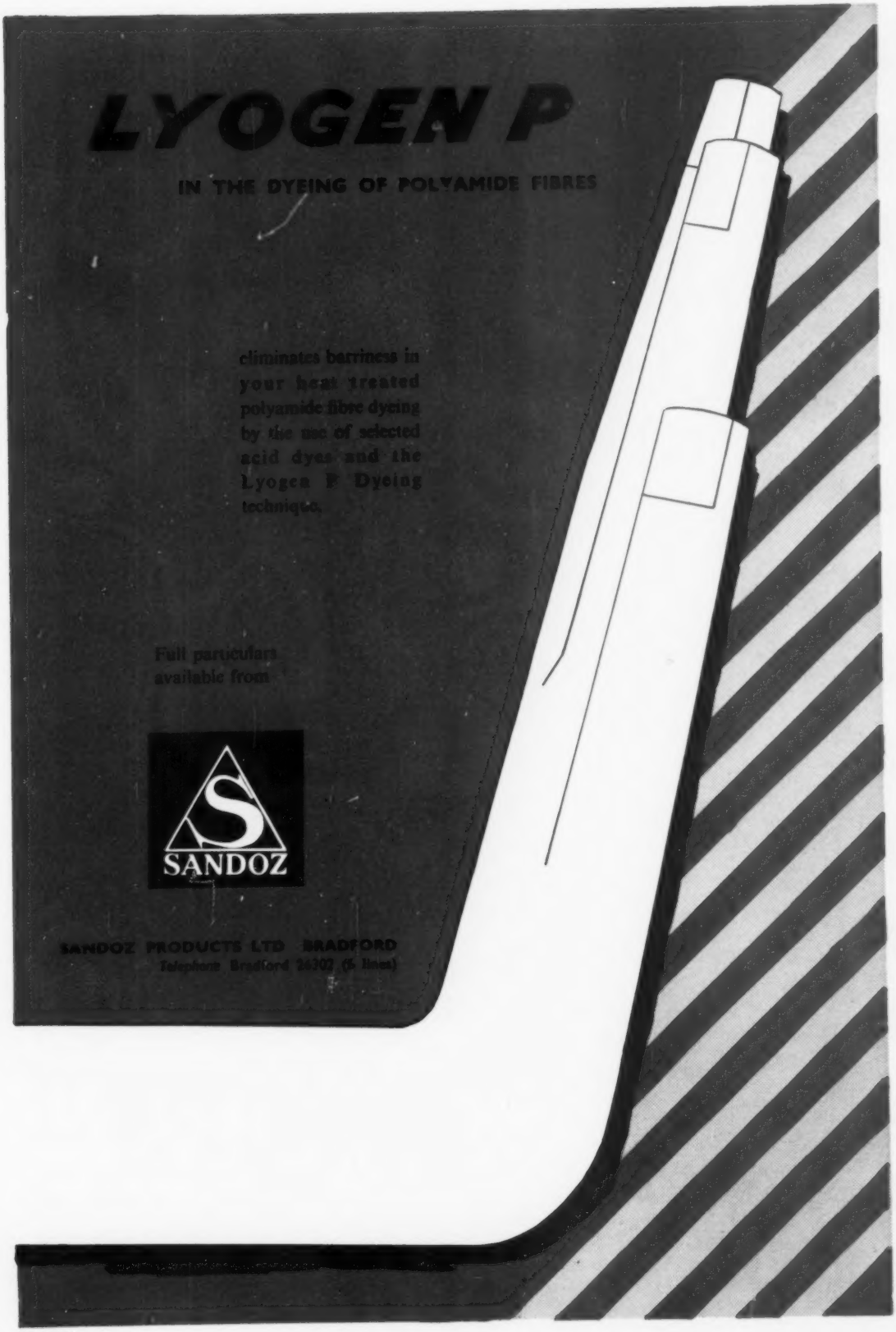

face centre

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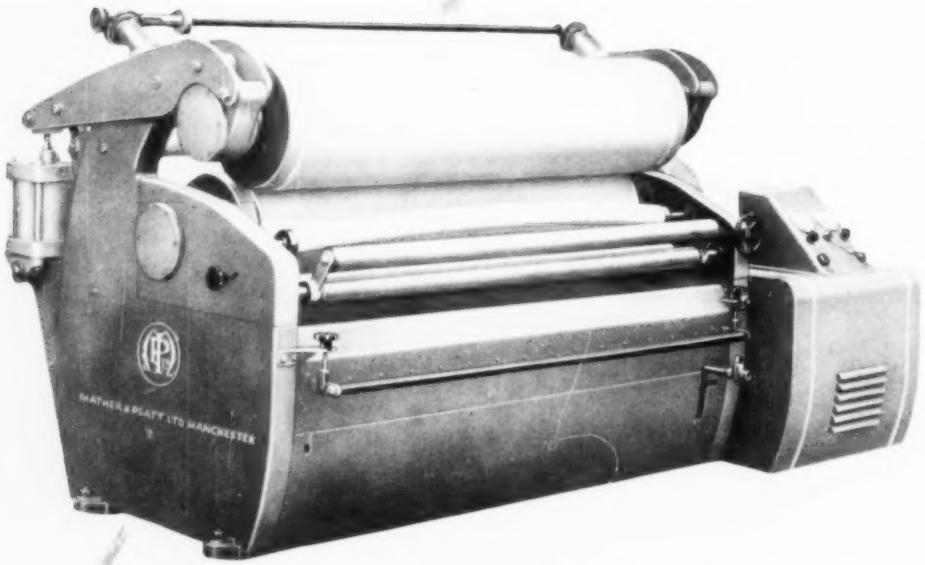

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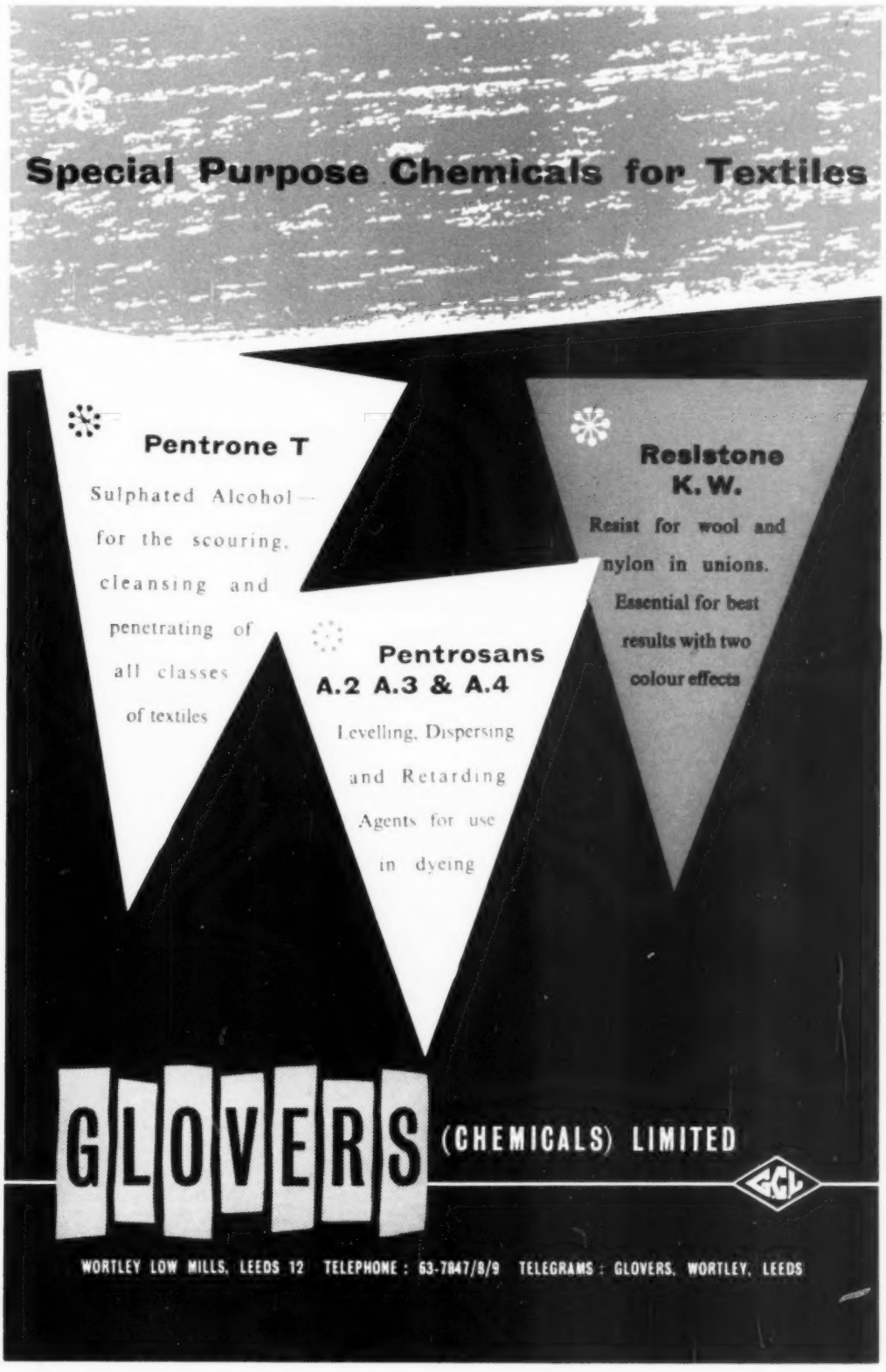
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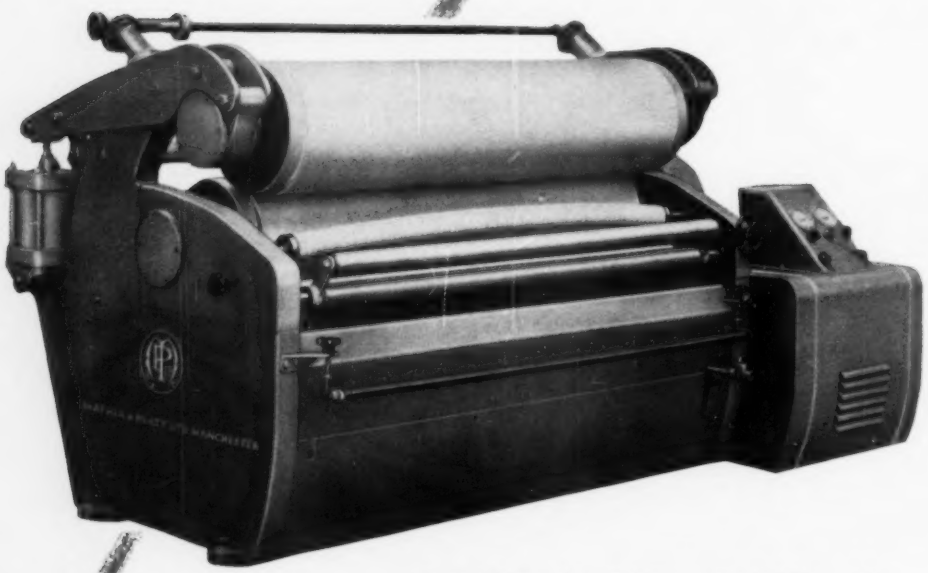

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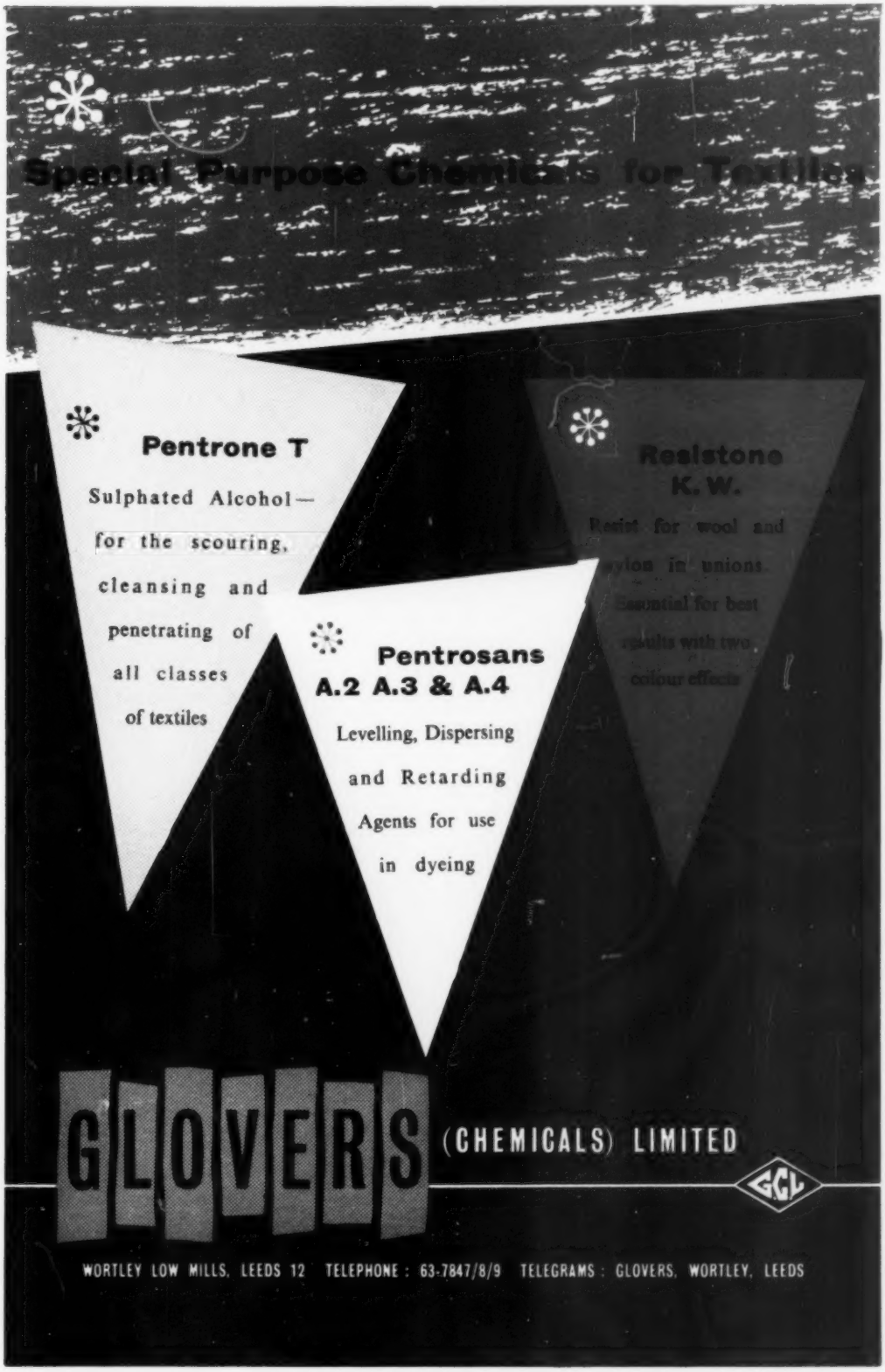
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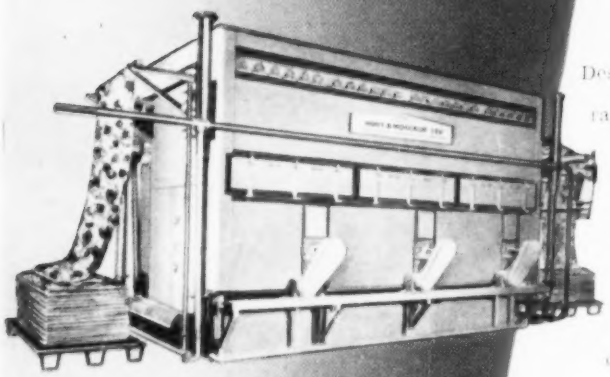

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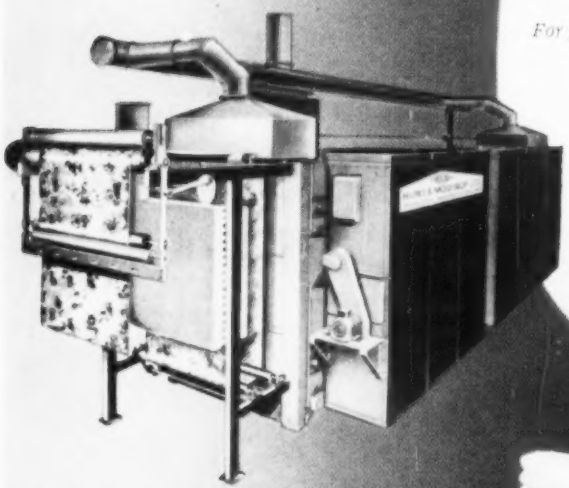

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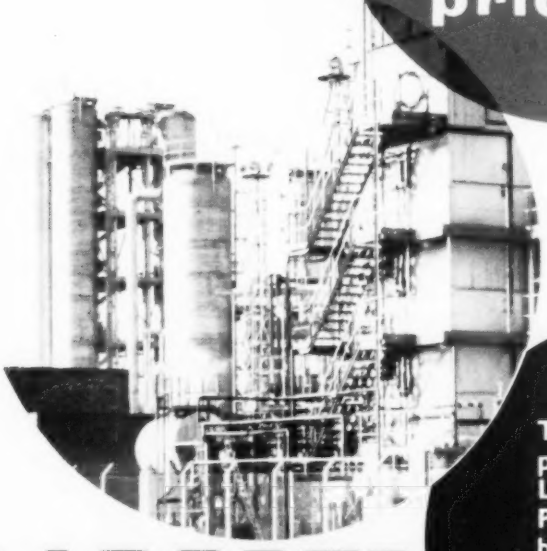

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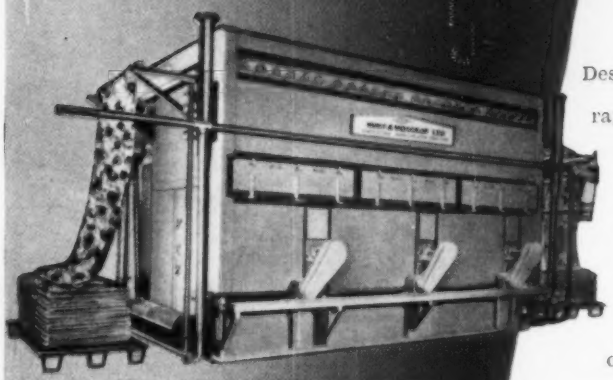

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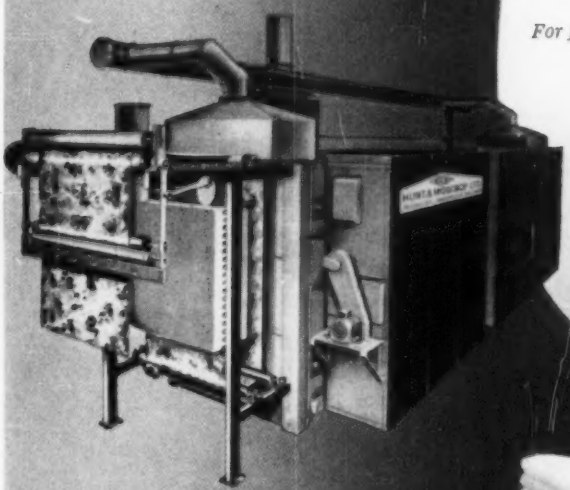

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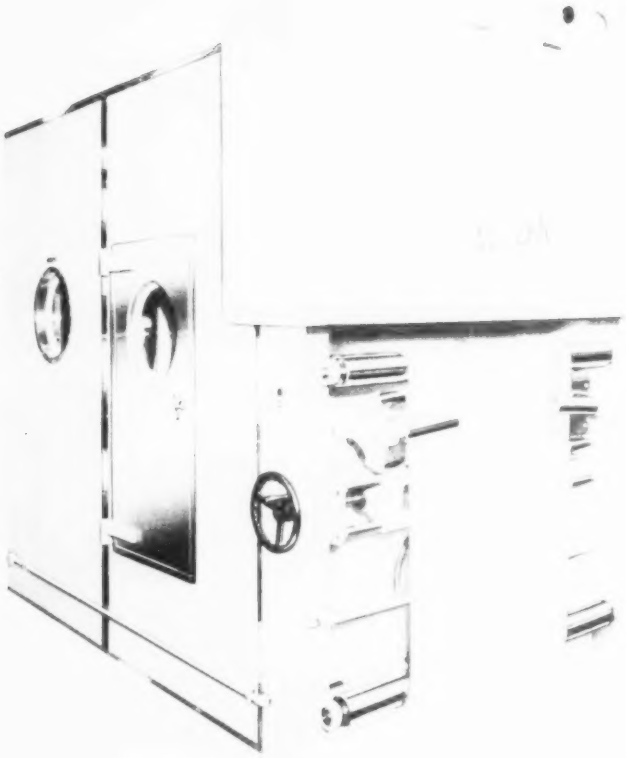

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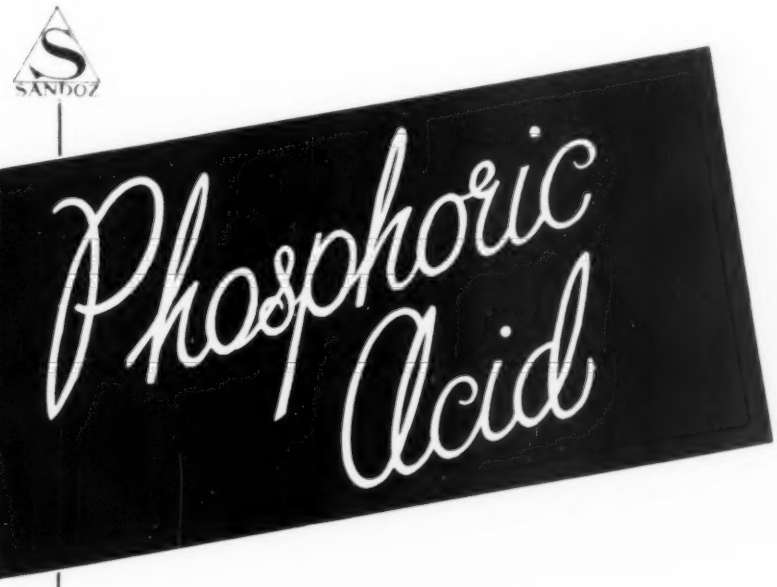

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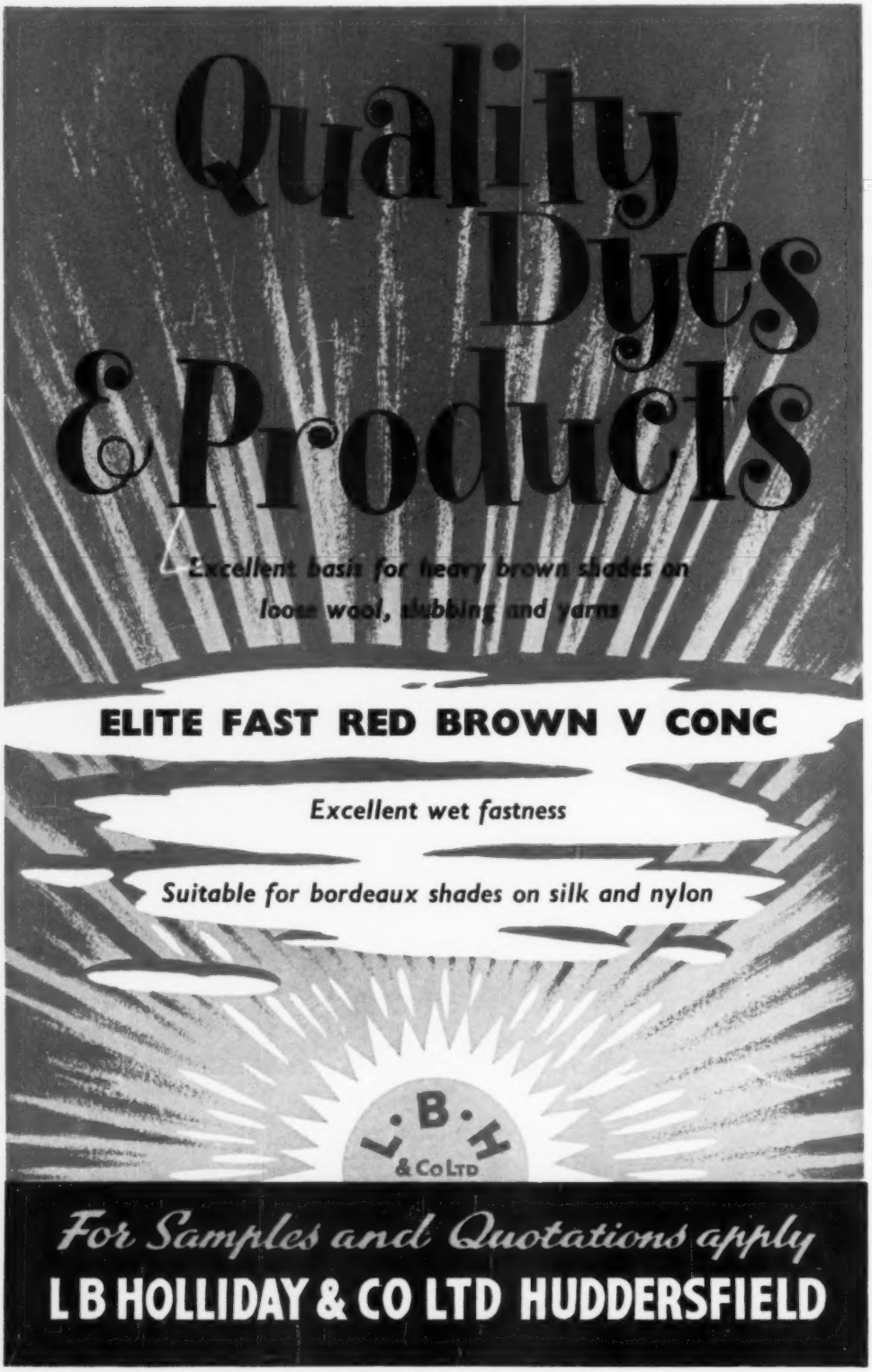
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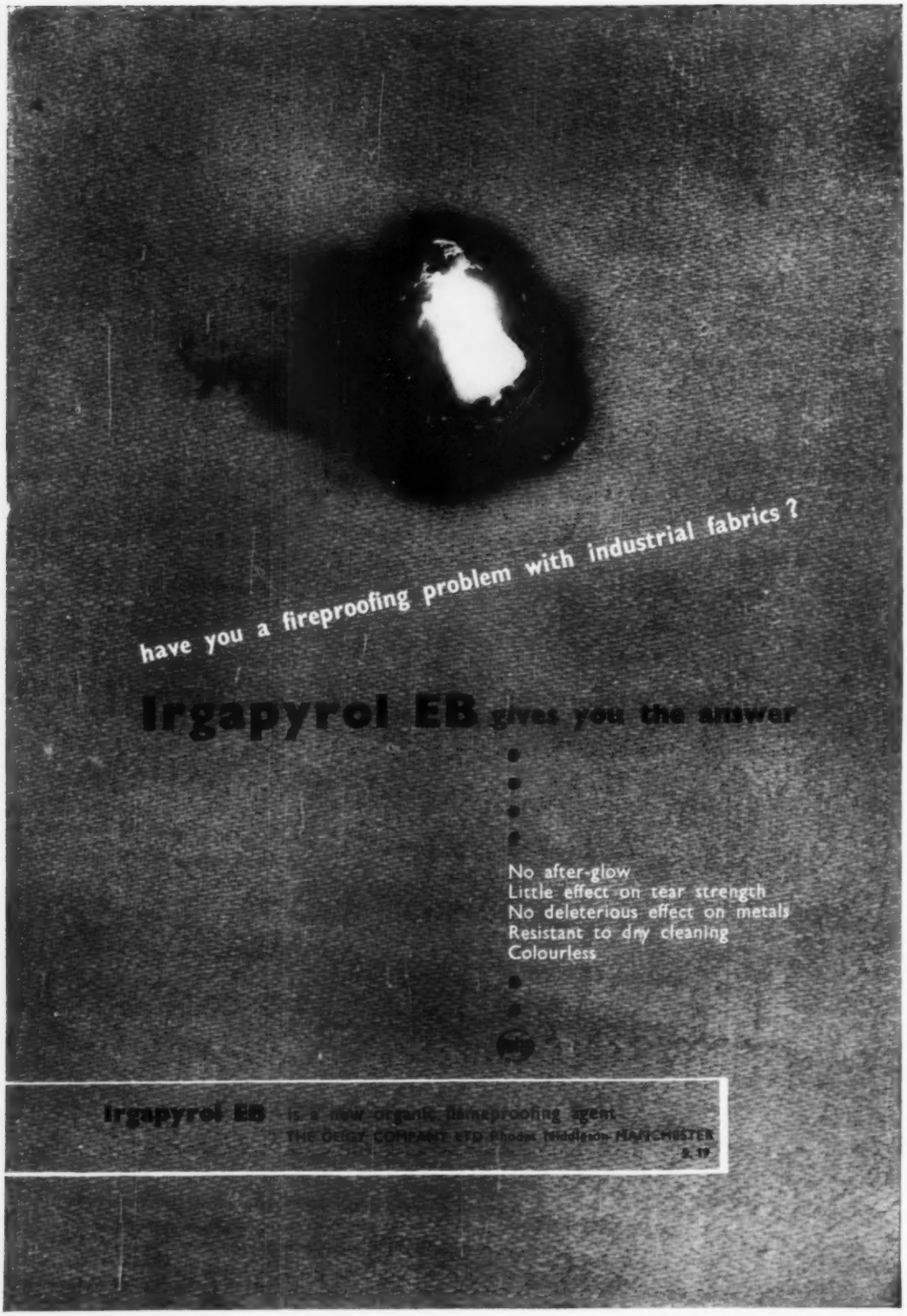
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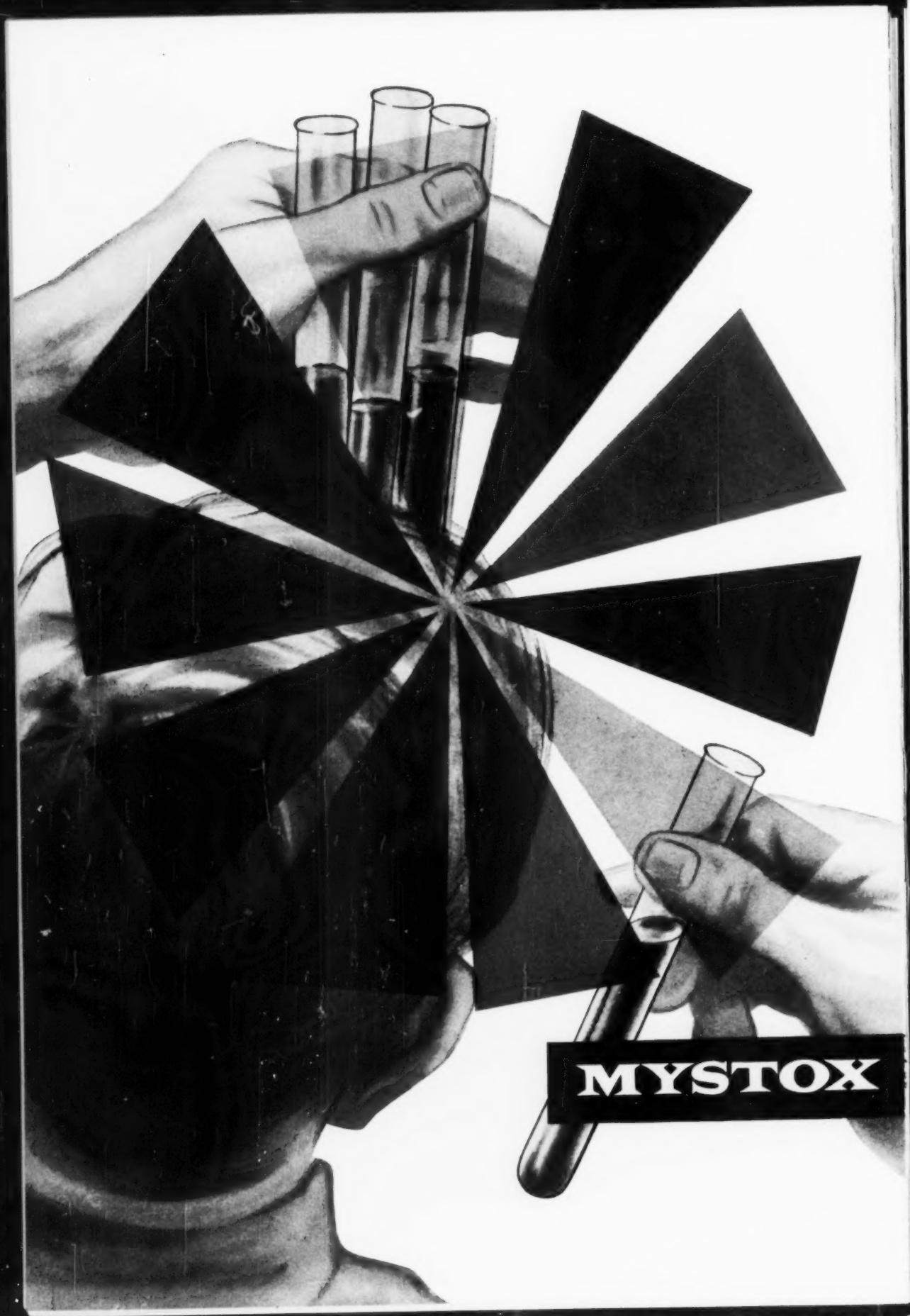
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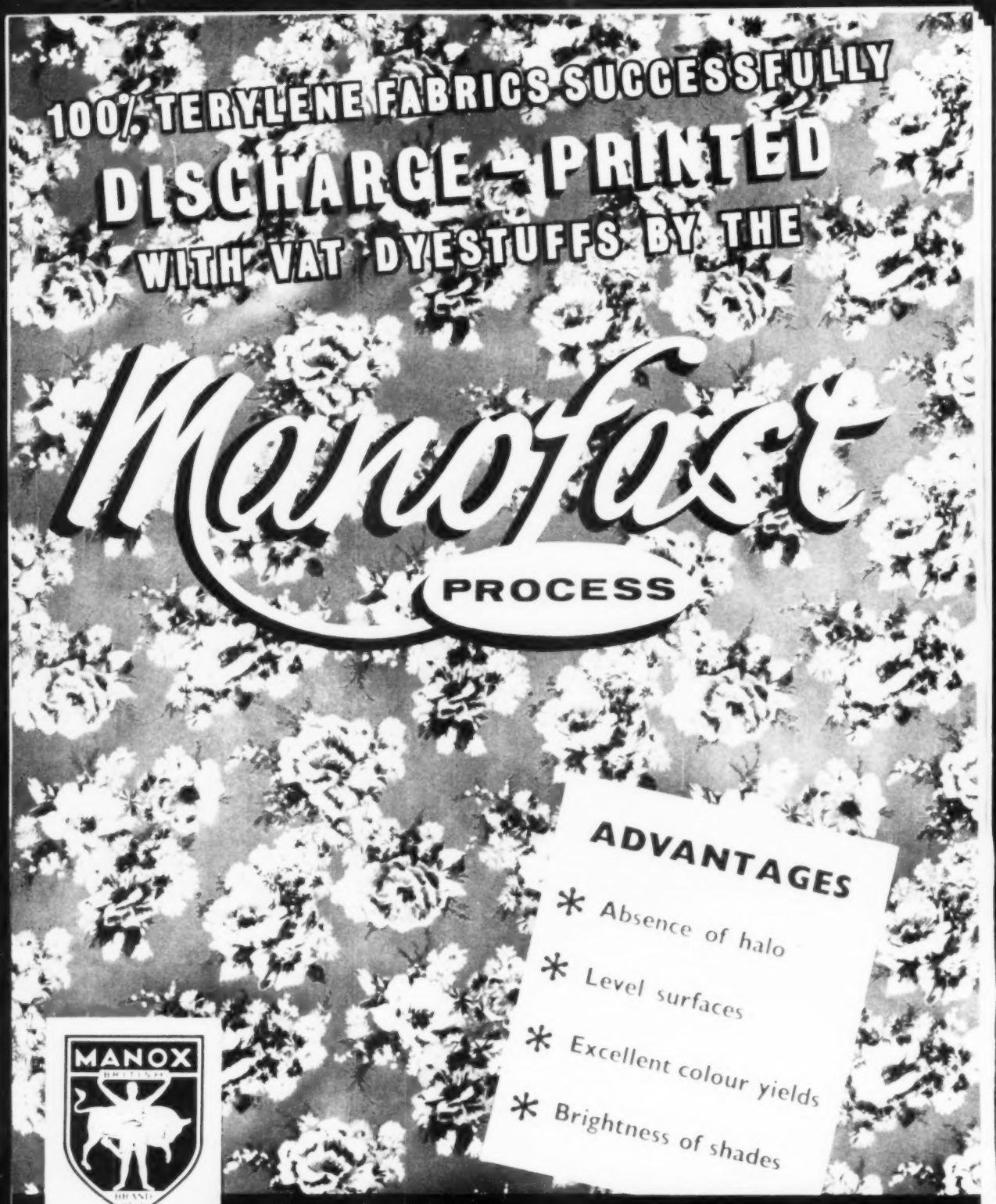

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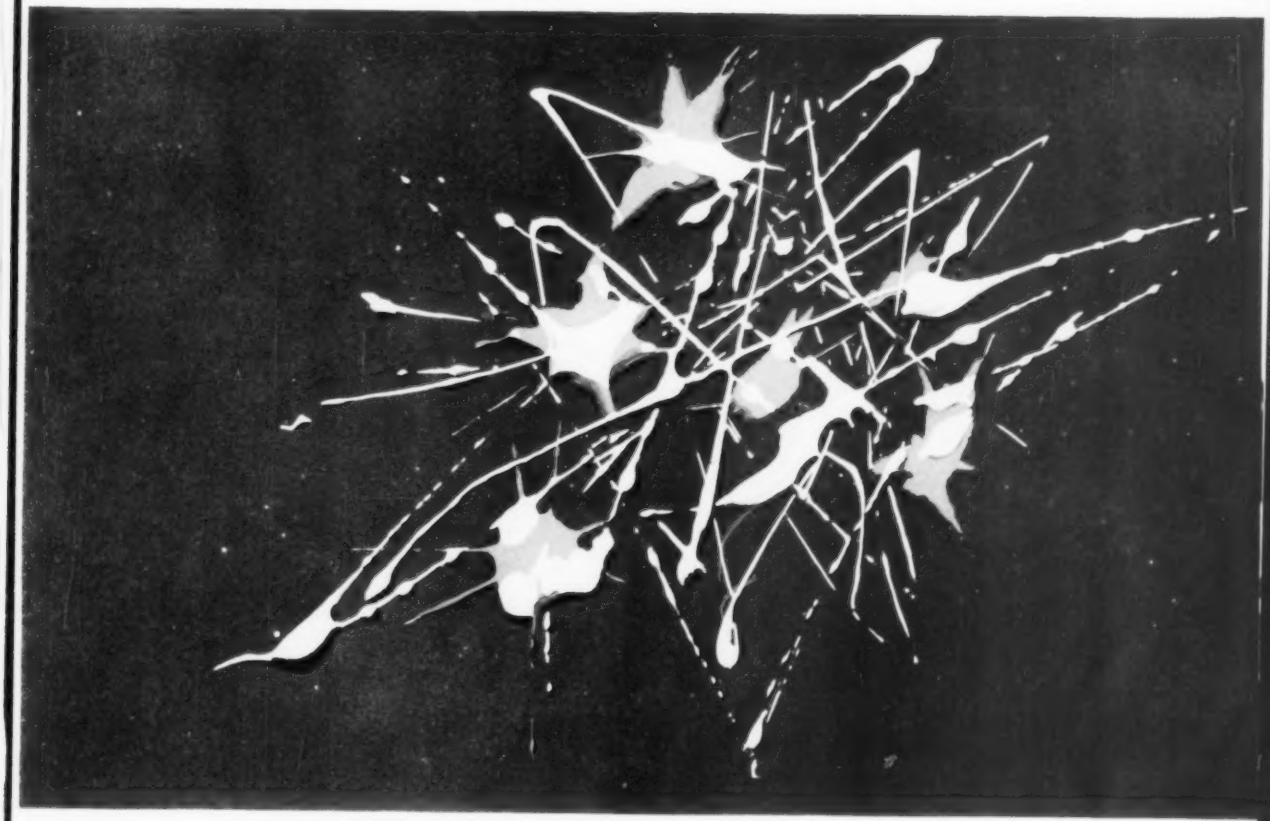

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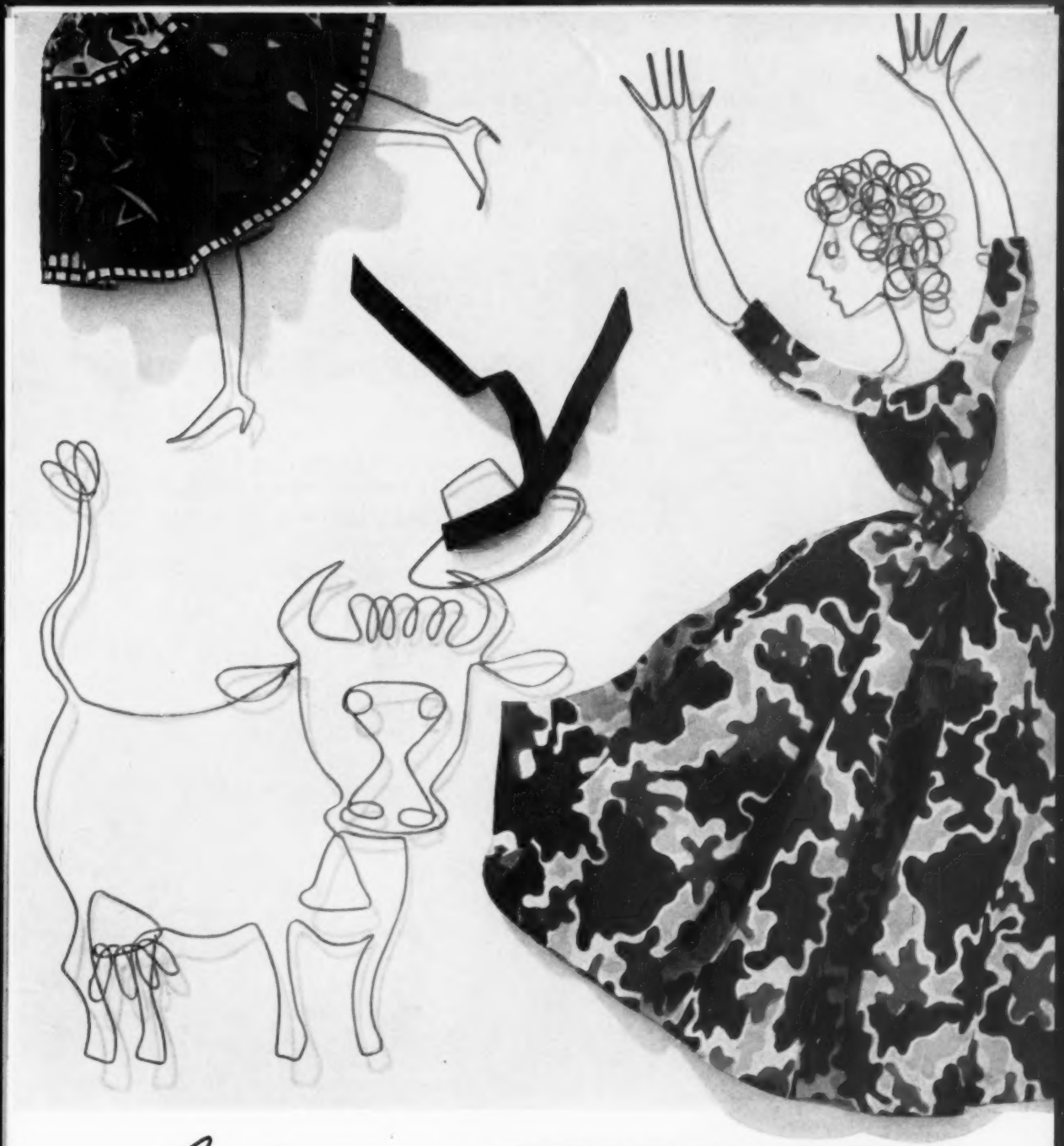

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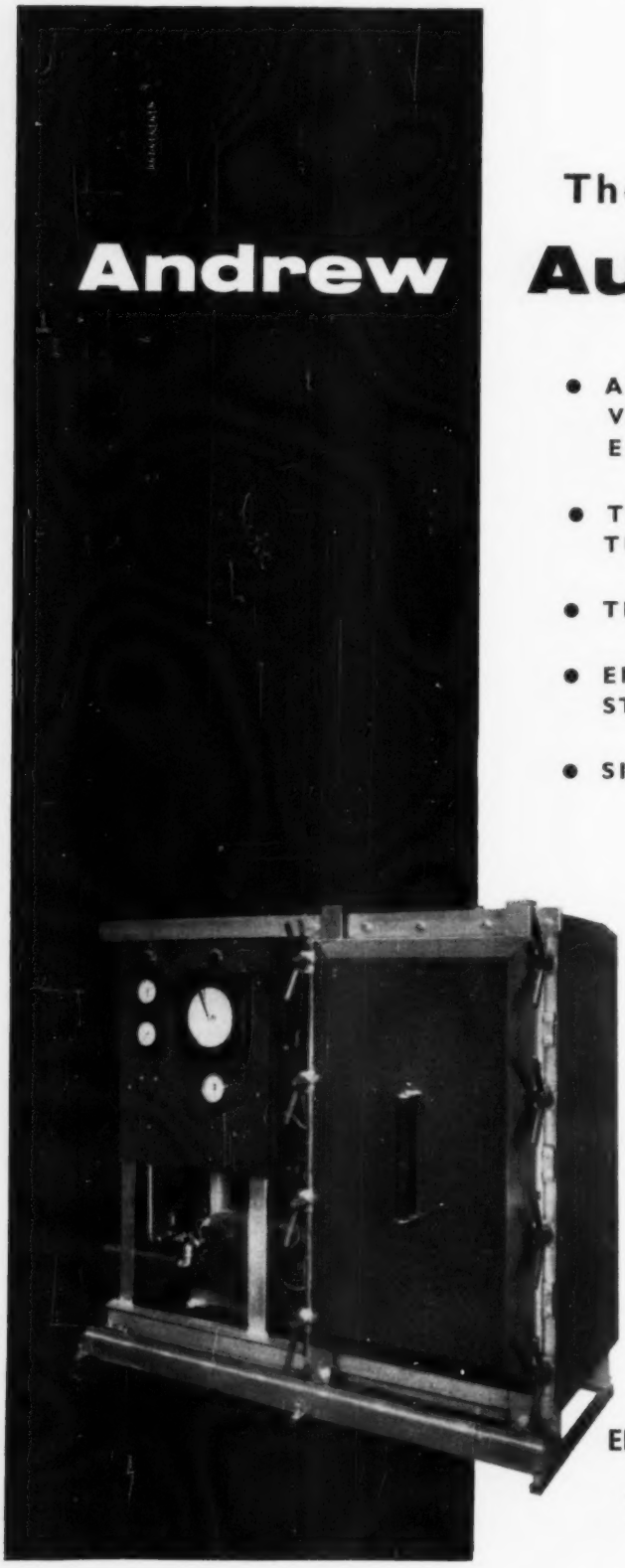

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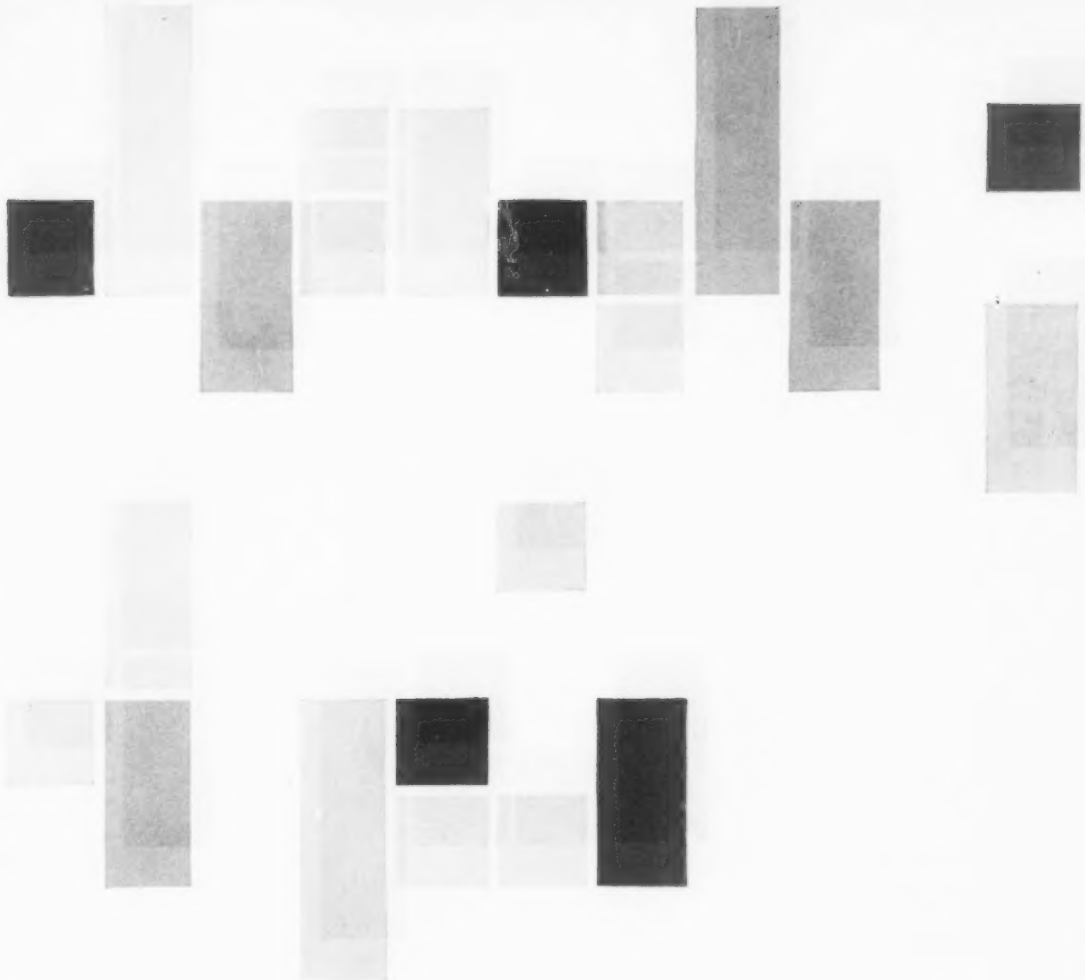

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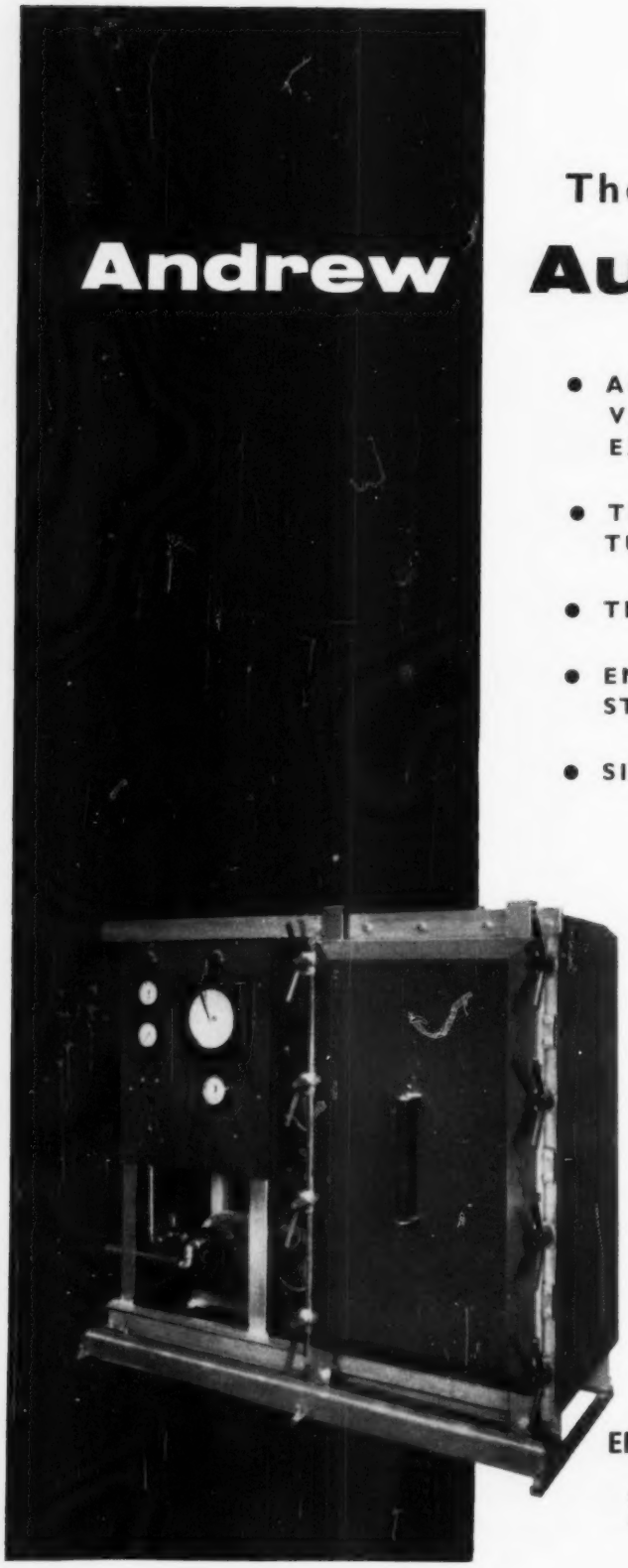

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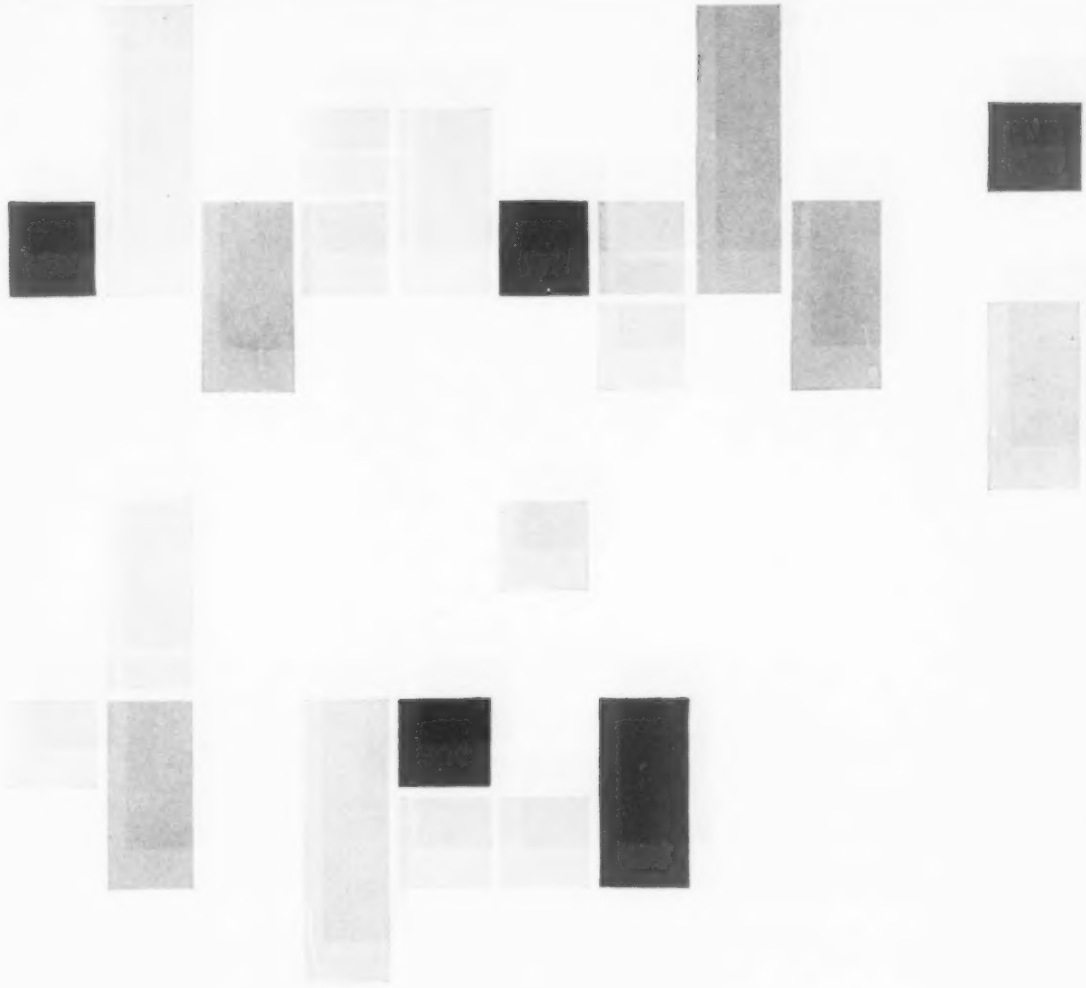

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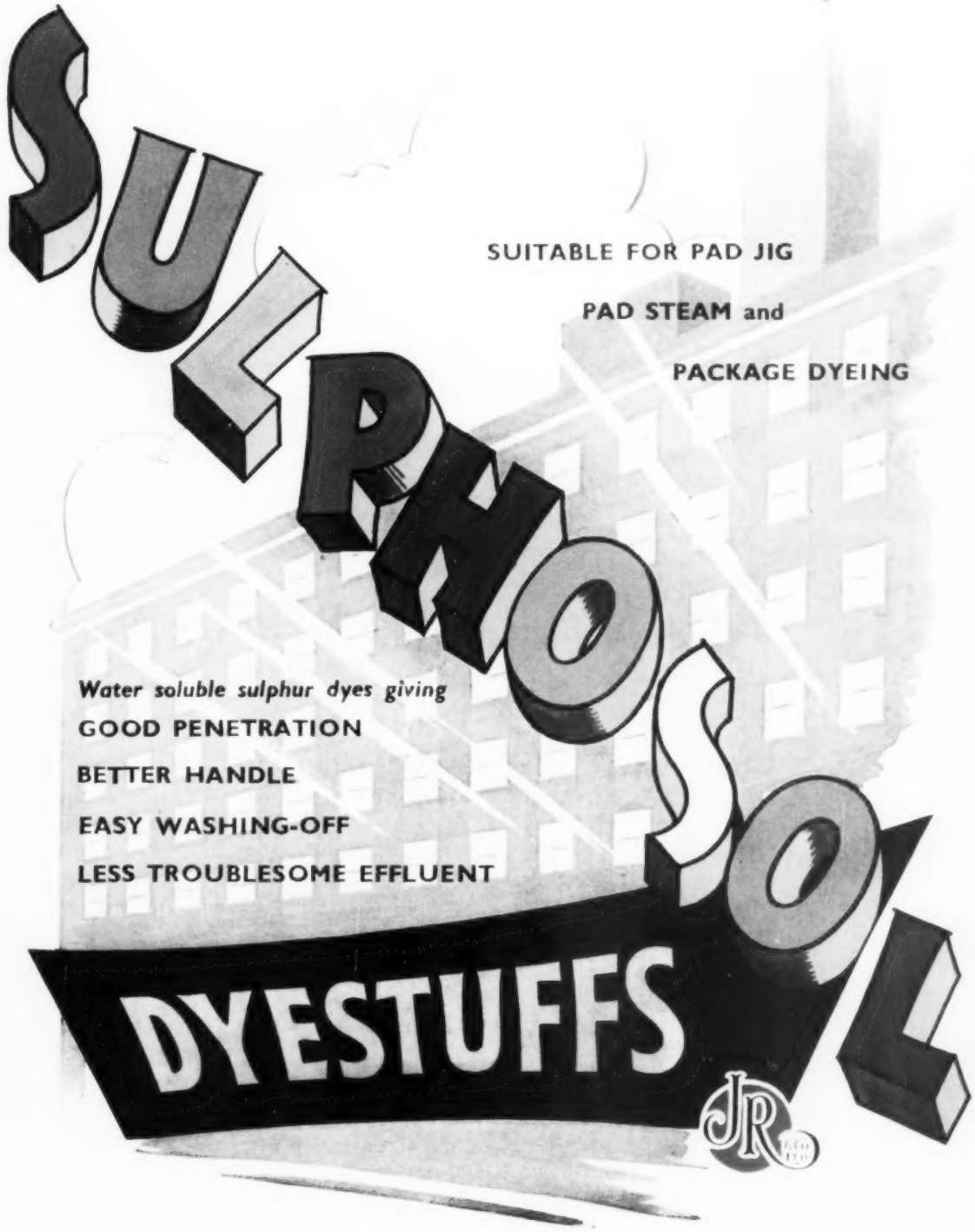

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Kinetic Isotope Effects and Isomeric Ratios for the Coupling of Deuterated 1-Naphthol-3-sulphonic acid
R. Ernst, O. A. Stamm, and H. Zollinger
Helv. chim. Acta, **41**, 2274–2278 (Dec. 1958)

Azo couplings of 4-chlorodiazobenzene with [2:4-di-D]-1-naphthol-3-sulphonic acid show an isotope effect (k_H/k_D = 3·1) which is smaller than that of the analogous reaction with 2-naphthol-6:8-disulphonic acid. These differences are explained as a function of k_{-1}, i.e. the rate of the reverse reaction of the first stage, and this constant is influenced by the different steric conditions in the intermediates of the reactions. In the coupling of [2:4-di-D]-1-naphthol-3-sulphonic acid an orientation isotope effect has also been found: the *o*/*p* ratio has a larger value, since the *para* coupling is more strongly base-catalysed than the *ortho* reaction, and consequently the kinetic isotope effect of the *para* coupling is also larger. H.H.H.

Preparation of Naphthylene-1,5-diamine
S. S. Pen'kovskiĭ and S. S. Kuznetsova
Zhur. priklad. khim., **31**, 955–957 (June 1958)

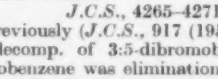
Fusion of naphthalene-1:5-disulphonic acid with 8·9 mol. of NaOH per mol. of acid at 310–330°c. during 3 hr. leads to the formation of 1:5-dihydroxynaphthalene. Charring in the reaction vessel, which is more apparent when heating is done with a naked flame, lowers the yield owing to decomposition, which is proved by the presence of naphthalene and H_2S. Treatment of the dihydroxy compound so obtained with 21·6% NH_4OH soln. in the presence of $(NH_4)_2SO_3$ soln. in a sealed tube at 125°c. leads to the formation of naphthylene-1:5-diamine.
T.Z.W.

Effect of the Nitro Group on the Reactivity of the Diphenyl Nucleus in Nitration. Competitive Nitration of Nitrodiphenyls and Benzene by Nitric Acid in Acetic Anhydride
Y. Mizuno and O. Simamura
J.C.S., 3875–3879 (Nov. 1958)

Competitive nitration of nitrodiphenyls and benzene by nitric acid in acetic anhydride at 0°c. gives proportions of isomeric dinitrodiphenyls and partial rate factors which show that the polar effect of the nitrophenyl groups is similar to that of halogen atoms with mesomeric electron-releasing and inductive electron-attracting effect. The theoretical implication of the change in the *ortho* : *para* ratio of the nitration products of isomeric nitrodiphenyls is discussed. H.H.H.

Thermal Decomposition of 3,5-Dibromobenzene 1,4-Diazo-oxide
M. J. S. Dewar and A. N. James
J.C.S., 4265–4271 (Dec. 1958)

It was deduced previously (*J.C.S.*, 917 (1958)) that the first step in the decomp. of 3:5-dibromobenzene 1:4-diazo-oxide in chlorobenzene was elimination of nitrogen and formation of the diradical (I), together with small amounts of diphenyl deriv. probably formed by interaction of I with the solvent. It is now reported that similar reactions occur in *o*- and *p*-chloro-, bromo-, and nitrobenzene, and, from the variation of the relative reaction-rate factors, it is confirmed that I is the reactive intermediate. The diphenyl type of reaction is found to predominate in benzene, fluorobenzene, anisole, *NN*-dimethylaniline, and benzonitrile, and can be made to do so in all cases by addition of 1% ethanol, which acts as a hydrogen-transfer catalyst; 3:5-dibromo-4-hydroxydiphenyl deriv. are then obtained in fair yields.



(I)

H.H.H.

Electronic Structure and Physicochemical Properties of Azo Compounds. IV— LCAO–MO Models of Phenylazoazulene
F. Gerson and E. Heilbronner
Helv. chim. Acta, **41**, 2332–2342 (Dec. 1958)

Simple LCAO–MO models for the electron system of phenylazoazulene have been calculated according to E. Hückel's approximation method, and the theoretical predictions for the transition energies and the oscillator strength of the two long-wave transitions of both azulene and phenylazoazulene are compared with the corresponding experimental values. H.H.H.

Synthesis of Fast Dyes

I— Synthesis of Direct Azo Dyes of the J-acid Series
T. Nishi, S. Ouchi, and Y. Abe
Bull. Yamagata Univ. Engr., **4**, 123–130 (1956)

Direct azo dyes having 4-amino-2-methyl-5-methoxyazobenzene-4′-sulphonic acid as the diazo component and CH_3CO, Bz, furfuroyl, cinnamoyl, *p*-tosyl, C_6H_5 or *p*-tolyl derivatives of J acid as coupling components were prepared and compared. They were all violets of good fastness to light and washing. Introduction of CH_3 and CH_3O groups into the two *ortho* positions of the two *para* azo groups of the middle benzene nucleus improved fastness to washing but caused deterioration in fastness to light. Introduction of an acyl and aryl group into the amino group of J acid improved fastness to light but did not affect fastness to washing. Acylation or arylation of the amino groups of J acid caused hypsochromical effect in the following order: CH_3CO < *p*-tosyl. The bathochromic effect was in the order Bz < furfuroyl < cinnamoyl < C_6H_5 < *p*-tolyl. Decrease of substantivity was in the order *p*-tosyl > CH_3CO and for increase was in the order Bz < furfuroyl < cinnamoyl or C_6H_5 < *p*-tolyl.

II— Synthesis of Direct Azo Dyes from *x*-t-Butyl-2-aminoanisole
T. Nishi, K. Takagi, and Y. Abe
Ibid., 119–128 (1957)

Direct azo dyes having 4-amino-2-*tert.*-butyl-5-methoxyazobenzene-4′-sulphonic acid as the diazo component and CH_3CO, furfuroyl, cinnamoyl, *p*-tosyl, C_6H_5 or *p*-tolyl derivative of J acid as the coupling component are bluish violets of fairly good fastness to light and washing. Introduction of *tert.*-butyl and CH_3O groups into the two *ortho* positions to two *para* azo groups of the middle benzene nucleus increased a bathochromic effect and caused deterioration in fastness to light. Introduction of an acyl or aryl group into the amino group of J acid improved the fastness to light. Acylation or arylation of the amino group of J acid gave a hypsochromic effect in the following order: CH_3CO < *p*-tosyl. The bathochromic effect was in the order: Bz < furfuroyl < cinnamoyl < C_6H_5 < *p*-tolyl. Decrease of substantivity was caused by substitution in the amino group of J acid in the order CH_3CO > *p*-tosyl and for increase was Bz < furfuroyl < cinnamoyl or C_6H_5 < *p*-tolyl.

III— Synthesis of Direct Azo Dyes of the J-acid Series
T. Nishi, Y. Abe, and S. Ouchi
Ibid., 129–138:
Chem. Abs., **52**, 21112 (25 Nov. 1958)

Direct azo dyes having 4-amino-2-methyl-4-amino-3-methyl- and 4-amino-3-methoxyazobenzene-4′-sulphonic acid as diazo components and CH_3CO, Bz, C_6H_5 or *p*-tolyl derivatives of J acid as coupling components have been made. Reds were obtained from J acid acetyl- and Bz-J acids and violets from aryl J acid, all had fairly good fastness to light and washing. The bathochromic effect increased in the order CH_3(in X-position) < CH_3 (in Y-position) < CH_3CO (in Y-position) (X-position = *ortho* to azo group of sulphanilic acid side; Y-position = *ortho* to azo group of J-acid side). Introduction of acyl or aryl into the amino group of J acid improved the fastness to both light and washing. Acylation or arylation of the amino group of J acid caused increase in hypsochromic and hypochromic effect in the order CH_3CO < Bz and in the bathochromic and hyperchromic effects C_6H_5 < *p*-tolyl. Introduction of CH_3CO into the amino group of the J acid decreased the substantivity, increase being caused in the order Bz < C_6H_5 > *p*-tolyl, decrease was also caused in the order CH_3 (in X-position) > CH_3 (in Y-position) > CH_3O (in Y-position). C.O.C.

Heterocyclic Azo Dyes by Oxidative Coupling
S. Hünig, H. Balli, K. H. Fritsch, H. Herrmann, G. Köbrich, H. Weiner, E. Grigat, F. Müller, H. Nöther, and K. H. Oette
Angew. Chem., **70**, 215–222 (1958):
Chem. Abs., **52**, 20163 (25 Nov. 1958)

Review, 48 references. C.O.C.

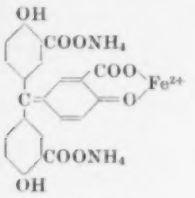
Covalently Bound Azo Dyes
R. R. McLaughlin
J.S.D.C., **74**, 857–858 (Dec. 1958)

Ferric Ammonium Aurintricarboxylate Chelate

A. K. Mukherji and A. K. Dey

J. Indian Chem. Soc., **35**, 237–242 (April 1958)

From pH measurements ferric ions form a 1:1 complex (I) with ammonium aurintricarboxylate. Spectrophotometric data at 25°c. give the values $k = 4{\cdot}8 \times 10^4$ at 25°c. and free energy of formation − 6·4 kcal./mole.



(I)

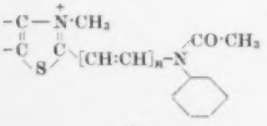
C.H.R.

Photographic Sensitisers. II—Mero-di- and -tri-carbocyanines

B. K. Patnaik and M. K. Rout

J. Indian Chem. Soc., **35**, 511–515 (July 1958)

The quaternary methiodides of 4-aryl-2-methylthiazoles and of 2-methylbenzothiazoles have been condensed with β-anilinoacrolein anil hydrochloride to give products of general formula (I) where $n = 2$ and with glutaconic aldehyde dianilide hydrochloride to give products (I) where $n = 3$. These have been condensed with 3-ethyl-2-thiothazolid-4-one to give the *mero*carbocyanine compounds—



(I)

C.H.R.

Absorption Spectra of Dyes. IV—Deformation of the Absorption Spectra of Some Copper-complex Dyes in Viscose Films by Dry Heat

Y. Ishi

Sen-i Gakkaishi, **14**, 718–723 (1958): *Chem. Abs.*, **52**, 21113 (25 Nov. 1958)

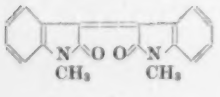
Trials were made with the direct blue dyes C.I. 24340, C.I. 24140, C.I. 24410 and Cu complexes derived from them. The maximum wavelength of the Cu complexes in solution was shifted toward the shorter wavelength side compared with the original dyes. The absorption spectra of viscose film dyed or treated with 1% aq. $CuSO_4$ after dyeing were measured as was the deformation of these spectra by dry-heating to 85°c. Increase in temperature reduced the absorption curve on the red side of the main absorption band. The rate of this deformation is accelerated by presence of NH_2 groups at *peri*-positions of OH groups, e.g. C.I. 24410. This deformation and change of colour of the dyed film is reduced by the $CuSO_4$ aftertreatment. The absorption curve was not deformed when the Cu complex of C.I. 24340 was used but it was slightly altered with the other two dyes. The colour change of films dyed with the complex dyes is similar to that of films dyed with the parent dyes and then treated with $CuSO_4$. C.O.C.

Oxidative Dimerisation of Oxindoles

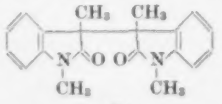
J. Harley-Mason and R. F. J. Ingleby

J.C.S., 4782–4783 (Dec. 1958)

1-Methyloxindole when oxidised with alkaline ferricyanide in a vigorously stirred two-phase system (water–carbon tetrachloride) gives (in poor yield) 1:1′-dimethyl*iso*indigo (I), while 1:3-dimethyloxindole gives (in fair yield) a mixture of the *mero* and racemic forms of 1:3:1′:3′-tetramethyl-leuco-*iso*indigo (II).



(I)



(II)

H.H.H.

Oxidation of Derivatives of *o*-Phenylenediamine. VI—Reductive Acylation of Anilinoaposafranines and Related Compounds

V. C. Barry, J. G. Belton, J. F. O'Sullivan, and D. Twomey

J.C.S., 4495–4498 (Dec. 1958)

Anilino*apo*safranines and related cpd. have their *p*-quinonoid system readily reduced by catalytic hydrogenation to give almost colourless cpd., which are rapidly reoxidised in air to their original state. In the presence of acetic or propionic anhydride, hydrogenation yields stable non-quinonoid cpd. containing 1–3 acyl groups.

H.H.H.

Triarylmethane Dyes

C. Albu

Anale univ. "C.I. Parhon" Bucureşti, Ser. ştiinţ. nat. (16) 89–98 (1957): *Chem. Abs.*, **52**, 21114 (25 Nov. 1958)

The absorption spectra of triarylmethane dyes shows a bathochromic effect for dyes containing a furan nucleus compared with those containing a benzene nucleus. This is caused equally by modifications of the alkyl radicals as well as to introduction of Br in the nucleus. Hypsochromic effect is seen in dyes containing a furan nucleus and it can be increased by changing the alkyl radical or by introducing Br into the nucleus. C.O.C.

Fluorescent Brightening Agents

D. A. W. Adams

J.S.D.C., **75**, 22–31 (Jan. 1959)

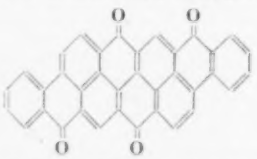
The nature of fluorescent brightening agents, their historical development, properties, and uses are reviewed, and some details are given of their application in soap and detergents, in textile processing, and in the production of paper. Their chemical constitution is then discussed, and indications are given of the effect on their properties of changes in structure. AUTHOR

4,10-Dibenzoylanthanthrone and its Cyclisation Products

N. S. Dokunikhin and L. A. Gaeva

Khim. Nauka i Prom., **3**, 280 (1958): *Chem. Abs.*, **52**, 20098 (25 Nov. 1958)

Account of an investigation to produce a polycyclic quinonoid vat dye. Naphthostyril treated with $AlCl_3$ and BzCl gave 6-benzoylnaphthostyril which, dissolved in hot aqueous NaOH yielded Na 8-amino-5-benzoyl-1-naphthoate. An alkaline solution of this product added in drops together with $NaNO_2$ to ice and HCl gave the diazonium compound which, reduced with ammoniacal Cu_2Cl_2 gave 4:4′-dibenzoyl-1:1′-binaphthyl-8:8′-dicarboxylic acid. This treated with conc. H_2SO_4 gave the bluish violet vat dye 4:10-dibenzoylanthrone which gives a bluish red with conc. H_2SO_4. Ring closure of this dye by oxidation with air, *m*-dinitrobenzene and *p*-nitrobenzoic acid at 130–170°c. in fused $AlCl_3$–NaCl gave—



which after crystallisation from fractional aqueous dilution of a H_2SO_4 solution and extraction with boiling 1-chloronaphthalene contained 2% Cl; substituting Na_2SO_4 and Na_2SO_3 for NaCl, reduced the Cl content to 1·4%. It dyes cotton green from a brown alkaline vat, but reddish violet from an acid vat followed by oxidation with H_2O_2 and prolonged ageing in air. C.O.C.

Pigmentation and Cell-wall Material of *Daldinia* Sp.

D. C. Allport and J. D. Bu'Lock

J.C.S., 4090–4094 (Nov. 1958)

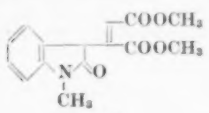
The chromogen of *D. concentrica* sporophores is found to be 4:5:4′:5′-tetrahydroxy-1:1′-dinaphthyl, and is the first 1:1′-dinaphthyl deriv. to be isolated from natural sources; it is oxidised enzymically to 4:9-dihydroxyperylene-3:10-quinone and to polymer. In the sporophores, these oxidation products cross-link the cell-wall material, apparently by combining with non-acetylated amino groups. H.H.H.

Michael Addition of 1-Methyloxindole and Dimethyl Acetylenedicarboxylate

J. A. Ballantine, R. J. S. Beer, and A. Robertson

J.C.S., 4779–4780 (Dec. 1958)

The title reaction, which occurs in the presence of sodium methoxide, affords two coloured cpd. which give almost identical analytical results, consistent with either of the expected products (I or II). From their ultraviolet absorption spectra these are regarded as different forms, possibly geometrical isomers, of dimethyl (1-methyl-3-oxindolylidene)succinate (II).



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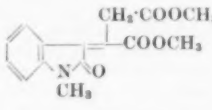


(II)

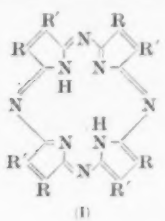
H.H.H.

Conjugated Macrocycles. XXXI— Catalytic Hydrogenation of Tetrazaporphins, with a Note on its Stereochemical Course

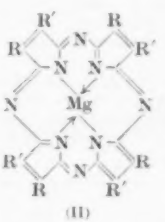
G. E. Ficken, R. P. Linstead, E. Stephen, and M. Whalley

J.C.S., 2879–3886 (Nov. 1958)

Controlled hydrogenations (with Pd black as catalyst) of tetrazaporphin (I: R = R′ = H), of its tetramethyl (I: R = H, R′ = CH_3), octamethyl (I: R = R′ = CH_3), and tetra*cyclo*hexeno (I: RR′ = $[CH_2]_4$) deriv., and of the corresponding Mg complexes (II) are described. With the more sol. Mg pigments in pyridine, preparative hydrogenation is practicable, whereas it is slow and accompanied by considerable loss with the metal-free pigments. After absorption of 2 mol. of hydrogen, royal-blue soln. are obtained which do not exhibit the strong visible and ultraviolet fluorescence characteristic of the parent pigments; the new blue pigments, either when dissolved or solid, are readily oxidised in air to the starting materials. In acetic acid, the reduced Mg pigments from the above subst. tetrazaporphins give purple metal-free cpd. which are reasonably stable to oxidation at room temp. The stereochemical course of the hydrogenation is discussed, and the dehydrogenation of tetra*cyclo*hexenotetrazaporphin with quinones is described.



(I)



(II)

H.H.H.

Analysis, Composition and Purification of Commercial Copper Phthalocyanine (C.I. 74160)

V. F. Borodkin

Izvest. Vyushikh Ucheb. Zavedenii, Khim. i Khim. Tekhnol., (2), 147–151 (1958); *Chem. Abs.*, **52**, 20774 (25 Nov. 1958)

To determine copper phthalocyanine dissolve 1 g. in 12 g. H_2SO_4 at 15–20°c., filter on a previously weighed funnel and paper, wash with water at > 80°c., dry and weigh. This gives the pure copper phthalocyanine. The commercial product obtained by the wet "baking process" is stronger than that yielded by the dry "baking process". Purification of commercial copper phthalocyanine with water and HCl and recrystallisation from H_2SO_4, still leaves it with some coloured impurities which are insoluble in dilute mineral acid. Unlike copper phthalocyanine they are soluble in organic bases, e.g. pyridine, quinoline, dimethylaniline and aniline. When commercial copper phthalocyanine (5 g.) was mixed with aniline (30 g.) at 50°c. for 3 hr., filtered and the insoluble residue on the filter washed with aniline 10 ml., no copper was found in the filtrate. Analysis of commercial copper phthalocyanine showed that it contained 2·5–3·2% of impurities. C.O.C.

Carotenoids and Related Compounds

C. K. Warren and B. C. L. Weedon

VI— Some Conjugated Polyene Diketones, and their Comparison with Capsorubin

J.C.S., 3972–3986 (Nov. 1958)

VII— Synthesis of Canthaxanthin and Echinenone

Ibid., 3986–3993

Carotene (C.I. Natural Yellow 26) Concentrates from Carrots in the Poznam Food Products Manufacturing Co.

Z. Pazola and J. Wenta

Przemysl Spożywczy, **11**, 439–441 (1957)

Carrots are squashed at 120 atm., the juice brought to pH 4·5–4·8 and 90°c. The coagulate of protein and carotene is filtered off and thrice extracted with cocoa butter or beef tallow at 60°c. for 60 min. with vigorous stirring. The fat is then filtered in steam-heated hydraulic presses to give a clear concentrate containing 200–300 mg. % of carotene. The stability of the carotene is increased by adding 1% wild rose extract containing 200 mg. % tocophenol. C.O.C.

Chemistry of Carmine (C.I. Natural Red 4) and of its Solutions used in Microscopy

H. Harms

Naturwissenschaften, **44**, 327 (1957)

The elementary analysis of Carmine is complicated because of the presence of about 25% protein and variable amounts of water. It consists of the skeleton $H_2O{\cdot}AlO_2CaOAlO_2{\cdot}H_2O$ having 4 O about each chelated Al, these O being contributed by 4 carminic acid molecules (C.I. 75470). With $AlCl_3$ Carmine yields 7·6 HCl (theory 8) which agrees with the conversion of each of the peripheral OH groups of the carminic acid moieties to $-OAl^+OH$, each Al being also coordinately linked to the adjacent anthraquinone O, the product being mucicarmine. On standing dilute solutions of mucicarmine soon yield a red, Cl-free precipitate, probably the $-OAl(OH)_2$ analogue of mucicarmine. The capricious tendency of mucicarmine to stain either mucopolysaccharides or these together with nuclei is ascribed to the variation in ionic state of mucicarmine with pH. In the commonly used solution (pH 3·8) only the 4 peripheral Al atoms are cationic, while at lower pH the central Al atoms also become cationic. The central Al atoms are also cationic at pH 1·95. Peripheral chelation is also involved in the ready solubility of Carmine in solutions of alums to yield bluish red to dark bluish black stains for nuclei. Although Carmine liberates little CO_2 from aq. $NaHCO_3$, it looses CO_2 by dry heat alone. Alkaline Carmine involves conversion of –Al-OH to anionic $-Al-O^-$ groups which when treated with HCl yield cationic $-Al^+{\cdot}OH$ moieties. C.O.C.

Paper-chromatographic Study of Numerous Acid Dyes. II

J. Kolsek

Chemiker-Ztg., **82**, 457–461 (1958); *Chem. Abs.*, **52**, 21114 (25 Nov. 1958)

Over 60 acid dyes have been separated into their components by paper chromatography using as solvent methanol–amyl alcohol–benzene–water (31 : 15 : 46 : 8 by volume) and paper No. 202 (C. Binzer, Hatzfeld–Eder). C.O.C.

Ferric–Aluminium Reds

M. Kranz

Przemysl Chem. **37** 349–352 (1958); *Chem. Abs.*, **52**, 21155 (25 Nov. 1958)

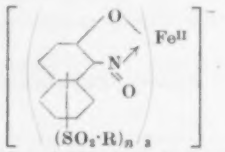
Best results were got by mixing aqueous ferric and aluminium sulphates with less than the stoichiometric amount of aqueous Na_2CO_3. The basic carbonates are precipitated as fine particles of red pigment. $Fe_2(SO_4)_3$ (15 ml. of 0·0071 g./ml.) were mixed with $Al_2(SO_4)_3$ (40 ml. of 0·0051 g./ml.) and Na_2CO_3 (5·5 ml of 0·0093 g./ml.) quickly added. The precipitate was filtered off, washed with water and calcined. It was a dark red pigment and with the Lovibond–Schofield Tintometer gave the following readings in international Lovibond units: red 11·8, yellow 4·3, and blue 2·9; after recalculating for pure colour the grey was 2·9, pure red 8·9, and pure yellow 1·4. The s.g. was 5·12 and alkalinity nil. It is suggested that the method can find application in the manufacture of composite ferric reds. 17 references. C.O.C.

PATENTS

α-Nitroso-β-naphthol Dyes

BASF *BP* 805,548

Green dyes for animal or synthetic polyamide fibres contain the anion—

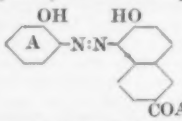


(R = Alk, aralkyl, *cyclo*Alk, Ar, heterocyclic radical, NH_2 where < 1 R in each mol. is other than NH_2, primary or secondary amine, carboxylic or sulphonic acid imide, alcohol or phenol; n = 1–2; II = valency of the bound Fe). They are made by reaction of Fe-yielding compounds with *o*-hydroxynitrosonaphthalenes containing 1–2 SO_2·R groups. Thus, 1-nitroso-2-hydroxynaphthalene-6-sulphonic acid dimethylamide is added to water (2000), $FeSO_4$,$7H_2O$ (16), potassium sodium tartrate (20), and a condensation product (100) of 1 mol. of HCHO with 2 mol. of naphthalene-2-sulphonic acid. Whilst stirring, Na_2CO_3 (17) is added at 60–70°C., and the dye separation is then completed by adding NaCl (300). The products have good fastness to moisture and light on wool and show considerably greater affinity than those from the *o*-hydroxynitrosonaphthalenes described in *GP* 869,103. E.T.

Red, Violet, and Blue Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, etc.

ICI *BP* 805,560

The metal complexes containing 1 atom of Co or Cr to 2 mol. of *oo'*-dihydroxymonoazo compounds—

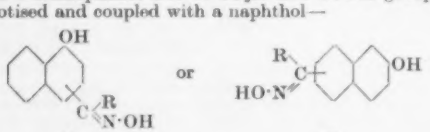


(ring A may carry substituents other than SO_2H or COOH; the group COAlk may be in the 3- or 8- instead of in the 6-position) dye wool, nylon, etc., from neutral or weakly acid baths. Some of the less soluble products are also suitable for cellulose acetate and for colouring lacquers, etc. Thus the monoazo compound 2-aminophenol-4-sulphonamide → 6-acetyl-2-naphthol is heated at 95–100°C. with chromous acetate in aq. ethylene glycol. The product dyes wool violet from a bath containing ammonium acetate. E.S.

Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc. having a Ketoxime Group

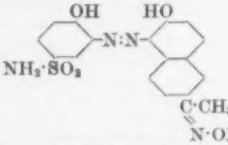
ICI *BP* 806,657

An *o*-aminophenol free of SO_2H and COOH groups is diazotised and coupled with a naphthol—



(I) (II)

(R = a hydrocarbon radical; the oxime group is in position 3 or 4 in formula I, and in position 6 or 8 in formula II) to give monoazo compounds whose 1:2 Cr(or Co)-complexes are mainly violet dyes for wool, nylon, etc., applicable from a neutral or weakly acid bath. The oximes are prepared from the corresponding ketones by heating with hydroxylamine in a suitable solvent. Thus diazotised 2-aminophenol-4-sulphonamide is coupled with methyl-2-hydroxynaphthyl-6-ketoxime to give—

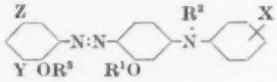


Heating in ethylene glycol with chromous acetate gives the violet chromium-complex. E.S.

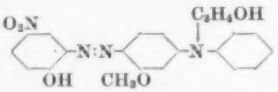
Metallisable Monoazo Disperse Dyes

Eastman Kodak Co. *USP* 2,843,580

Diazotised *o*-aminophenol and derivatives thereof are coupled with 3-hydroxydiphenylamine and derivatives thereof to give disperse dyes of general formula—



(R^1 = H, Alk or hydroxyalkyl; R^2 = H, Alk, hydroxyalkyl, or cyanoalkyl; R^3 = H or Alk; X = H, Hal, Alk, or O Alk; Y and Z = H, Hal, NO_2, Alk, haloalkyl or SO_2·NH_2) which may be metallised in substance or on the fibre. For aftertreatment on cellulose acetate, steaming after padding with nickel thiocyanate is the preferred process. The coupling components are prepared by heating 3-hydroxydiphenylamines in an autoclave with ethylene oxide. Thus 3-methoxydiphenylamine gives the *N*-β-hydroxyethyl derivative, which with diazotised 2-amino-4-nitrophenol couples to give—



which dyes cellulose acetate orange of poor fastness to light. Padding the dyeing with 2–5% nickel thiocyanate, followed by drying and steaming for 10 min., gives a red of good light fastness. Metallisation in substance, by heating the monoazo compound in *O*-methyl ethylene glycol with nickel acetate gives a complex which dyes nylon, wool, or polyacrylonitrile deep red. E.S.

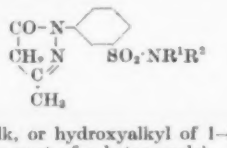
Yellow Metal-complex Monoazo Pyrazolone Dyes for Wool and Nylon

General Aniline *BP* 806,668

The Cr complexes of monoazo dyes obtained by coupling diazotised anthranilic acid (or its dihalogeno derivatives) with a 1-dichlorophenyl-5-pyrazolone, are yellow dyes applicable to wool and nylon from neutral baths. Thus diazotised anthranilic acid is coupled with 1-(2′:5′-dichlorophenyl)-3-methyl-5-pyrazolone, and the product heated at 125°C. in formamide with chromium formate. The Cr-complex so formed is mixed with a dispersing agent before drying.

BP 806,678

Similar dyes are obtained by converting into their Cu, Ni, Fe, Co, or Cr complexes the monoazo compounds from diazotised anthranilic acid coupled with 1-(*m*-sulphamylphenyl)-3-methyl-5-pyrazolones—

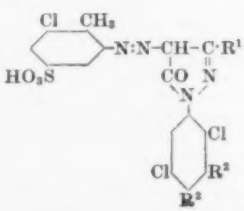


(R^1, R^2 = H, Alk, or hydroxyalkyl of 1–4 C, or R^1 and R^2 together form part of a heterocycle). E.S.

Yellow Monoazo Pyrazolone Acid Dyes

General Aniline *USP* 2,842,537

Yellow dyes—

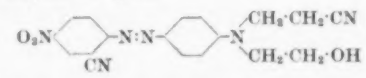


(R^1 = lower Alk, COOH, or COO Alk; one R^2 = H, the other = SO_3H) have better washing fastness on wool than existing similar monoazo pyrazolone yellows. In the preferred dyes the R^2 in the *p*-position is SO_3H, the other R^2 is H. E.S.

Red and Violet Monoazo Disperse Dyes having Cyano Groups

ICI *BP* 806,271

2-Cyano-4-nitroanilines and their 6-halogeno derivatives are diazotised and coupled with alkylanilines which carry an *N*-β-cyanoethyl group, to give disperse dyes faster to light than the similar dyes of *BP* 447,561 (J.S.D.C., **52**, 404 (1936)) which do not have the *N*-β-cyanoethyl group. Thus 2-cyano-4-nitroaniline, diazotised in nitrosyl-sulphuric acid and coupled with *N*-β-cyanoethyl-*N*-β′-hydroxyethylaniline gives—

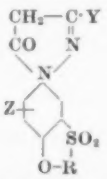


which dyes cellulose acetate bluish-red. E.S.

Yellow to Red Metal(Chromium and Cobalt)-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.

FBy *BP* 806,709

Pyrazolones of general formula—



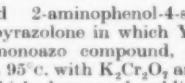
(Y = Alk, Ar, CO·NH$_2$, CO·NH Alk, CO·N (Alk)$_2$, or COO Alk; Z = H, Alk, Hal, etc., but not SO$_3$H or COOH: R = CH$_2$ or *o*-arylene) are coupled with diazotised *o*-aminophenol or anthranilic acid, or their derivatives free of SO$_3$H or COOH groups, and the products converted into metal complexes containing 1 atom of Co or Cr to 2 mol. of monoazo compound, to give the title dyes. The pyrazolones used are prepared from suitable heterocyclic ether sulphones containing NH$_2$ groups (whose preparation is described in *BP* 753,261) by diazotising and condensing with acetoacetic esters, benzoylacetic esters, or oxalacetic esters.

Thus diazotised 2-aminophenol-4-sulphonamide is coupled with the pyrazolone in which Y = CH$_3$, Z = H, R = CH$_2$. The monoazo compound, dissolved in aq. NaOH, is stirred at 95°c. with $K_2Cr_2O_7$ and glucose to give the Cr-complex, which dyes wool reddish-orange from a formic acid bath. E.S.

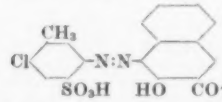
Monoazo and Diazo Dyes and Lakes from Hydroxy-alkylamides of 3,2-Hydroxynaphthoic Acid

BASF *BP* 806,721

Hydroxyalkylamides of 3:2-hydroxynaphthoic acid—



(R^1 = H or β-hydroxyalkyl; R^2 = β-, γ-, or δ-monohydroxy-, or β:γ-dihydroxy-alkyl of < 5 C) are coupled (*a*) with diazotised sulphonic acids of aniline or naphthylamine, or tetrazotised diaminosulphonic acids of the diphenyl or diphenylmethane series, to give monoazo and disazo dyes whose Ca and Ba lakes are pigments for lacquers and varnishes, and (*b*) with similar diazotised or tetrazotised amines having *o*-OH, *o*-OAlk, or *o*-COOH groups to give metallisable dyes for wool and nylon. Thus methyl 3:2-hydroxynaphthoate heated with monoethanolamine gives 3-hydroxy-2-naphtho-β-hydroxyethylamide (I) which is coupled with diazotised 3-amino-6-chlorotoluene-4-sulphonic acid to give—



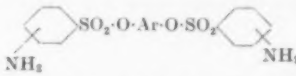
whose Ba lake is a bright red pigment of good oil fastness.

Diazotised 2-amino-4-chlorophenol-6-sulphonic acid coupled with I gives a blue monoazo dye applicable by either the afterchrome or metachrome process. E.S.

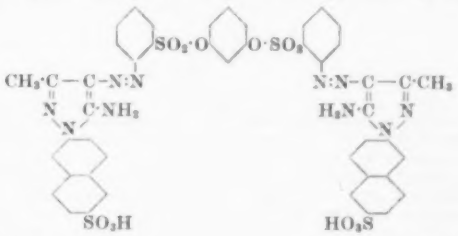
Yellow and Red Disazo Pyrazole Acid Dyes

FBy *BP* 805,777

Diamines—



(substituents other than SO$_3$H or COOH may be present) are tetrazotised and coupled with 2 mol. of a 1-aryl-3-methyl-5-aminopyrazole coupling in the 4-position, or with 1 mol. of such a pyrazole and 1 mol. of an *o*-coupling naphthylamine, to give acid dyes applicable from neutral or weakly acid baths. The diamines are prepared by esterifying aromatic dihydroxy compounds with 2 mol. of a nitrobenzenesulphonyl chloride, followed by reduction. Thus reaction of resorcinol with 2 mol. of *o*-nitrobenzene-sulphonyl chloride followed by reduction gives a diamine, which on tetrazotisation and coupling with 2 mol. of 1-(6′-sulphonaphthyl-2′)-3-methyl-5-aminopyrazole gives—

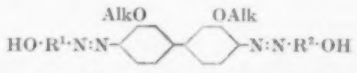


which is a yellow. E.S.

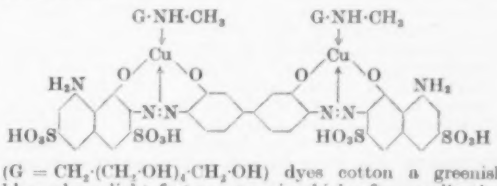
Metal(Copper)-complex Disazo Direct Dyes containing a Sugar-amino Group

DuP *USP* 2,842,536

Direct dyes of the type—



(R^1·OH and R^2·OH = residues of naphthol- and amino-naphthol-sulphonic acids), when coppered in presence of an amino-sugar CH$_2$·OH(CH·OH)$_n$·CH$_2$NHR^3 (*n* = 3 or 4; R^3 = H, CH$_3$, C$_2$H$_5$, or CH$_2$·CH$_2$·OH) give complexes of higher light fastness and improved nylon reserve than the similar products obtained by coppering in presence of ammonia, methylamine, ethylamine, ethanolamines, etc. Thus tetrazotised *o*-dianisidine is coupled in presence of NaOH with 2 mol. of H acid, and the product refluxed with ammoniacal CuSO$_4$ and *N*-methylglucamine. The complex formed—



(G = CH$_2$·(CH·OH)$_4$·CH$_2$·OH) dyes cotton a greenish blue whose light fastness remains high after application of resin finishes. E.S.

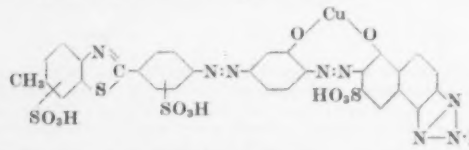
Metal-complex Disazo Dyes— Water-soluble Blacks for Inks

Electro Chemical Laboratories Corpn. USP 2,836,588

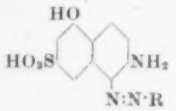
A diazotised aniline (or naphthylamine)-sulphonic acid (1·6–1·75 mol.) is coupled with a polyhydric phenol (1 mol.), and a soln. of FeCl$_3$ (or similar metal salt) is added, followed by pyridine, ammonia or other suitable base. The soluble complex so formed is precipitated by adding a water-soluble alcohol, and gives an intense black ink when dissolved in water. Thus diazotised sulphanilic acid (14 parts) is coupled with an ammoniacal soln. of catechol (3·5 parts), and a soln. of FeCl$_2$ is then added, followed by dropwise addition of pyridine. Isopropyl alcohol is then added, to produce a black, tarry precipitate, which on drying at 65°c. gives a friable solid. A 50% soln. of this in water may be used in hectographing. E.S.

Metal(Copper)-complex Disazo Direct Dyes having a Benzthiazole and a Triazole Residue
Ciba *BP* 806,050

Copper-complex disazo dyes of the type—



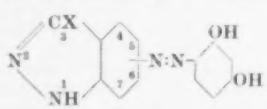
(R = benzene or naphthalene residue) give mainly greys on cellulose. They are made by coupling a diazotised dehydrothio-*p*-toluidinesulphonic acid (I) with *o*-anisidine (II) or a similar middle component, diazotising the aminomonoazo compound so formed, and coupling with a monoazo compound—



following by coppering under conditions which demethylate the *o*-anisidine residue and converting the *o*-aminoazo-R system into a triazole ring. Thus I is diazotised and coupled with the *ω*-methylsulphonic acid of II, and the product hydrolysed to the aminomonoazo compound, which is then diazotised and coupled with the acid-coupled monoazo compound metanilic acid → J acid. Stirring at 90–95°c. with ammoniacal $CuSO_4$ in presence of ethanolamine gives the required complex (R = *m*-$C_6H_4{\cdot}SO_3H$) which dyes cotton greenish-grey. E.S.

Metal-complex Monoazo Indazoles— Azoic Coupling Components for Blacks and Browns
Fran *BP* 806,166

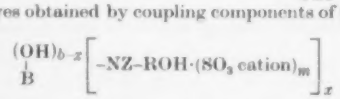
Diazotised 4-, 5-, 6-, or 7-aminoindazoles, which may be substituted in the 3-position by Hal, and in the 4, 5, 6, or 7 positions by Hal, NO_2, $SO_2{\cdot}NH_2$, or acylamino (this last group in only the *m*- or *p*-position to the NH_2 group), are coupled with resorcinol to give monoazo compounds—



(X = H or Hal), whose metal (especially Cr, Mn, Fe, Ni, and Cu) complexes give brown and black azoic dyes when coupled on the fibre with suitable diazo compounds. Thus 7-amino-4-chloroindazole is diazotised and coupled with resorcinol and the product refluxed with aq. $CoSO_4$ in presence of sodium tartrate. The metal complex so formed is dissolved in aq. alcoholic NaOH, mixed with the triazen obtained by condensing diazotised 2-amino-4-chlorotoluene with a suitable amine (e.g. *N*-*o*-carboxyphenylglycine) and a suitable thickener, and printed on cotton. After drying, neutral steaming produces a black. E.S.

Polymeric Azo Dyes for Use in Colour Photography
B. Gaspar and P. D. Dreyfuss *USP* 2,844,574

The dyes obtained by coupling components of formula—



(R = naphthalene residue; *m* = 1 or 2; Z = H or hydrocarbon; $B(OH)_{b-x}$ = *x* valent residue of a *b*-basic organic acid *x* being > 1 and *b* being ⩽ *x*) with polydiazo compounds are red, magenta, violet, blue, and cyan dyes which satisfy the highest requirements for multilayer photographic materials. Thus the dye obtained by coupling tetrazotised 1·3 mol. of 4:4′-diaminodiphenylether in aqueous pyridine with 1·0 mol. of *NN*′-adipyl-bis-(1-amino-8-hydroxynaphthalene-3:6-disulphonic acid) is a brilliant magenta. It is very soluble in water, fast to light and has no desensitising action. C.O.C.

Diazotisation of Arylamines
Dan River Mills *USP* 2,825,724

The arylamines *o*-aminoazotoluene, *p*-chloro-*o*-nitroaniline, *p*-aminoazobenzene, *o*-nitroaniline, and 2:5-dichloroaniline can be smoothly diazotised by dissolving in 1–1½ times their weight of diacetone alcohol containing a non-ionic dispersing agent, and pouring into a mixture of ice and aq. HCl to give a fine dispersion which reacts readily on addition of aq. $NaNO_2$. E.S.

Chlorination of Anthraquinone Derivatives
Ciba *BP* 807,092

Conventional chlorination methods suffer from such disadvantages as, e.g. (*a*) slow reaction on the large scale, (*b*) corrosion of plant, (*c*) inaccuracy in proportions of reactants due to the volatility of the chlorinating agent, (*d*) variability of product quality and, (*e*) contamination of the product with S when sulphuryl or thionyl halides and $AlCl_3$ are used. It is claimed that anthraquinone derivatives containing ⩽ 9 benzene rings (e.g. vat dyes or intermediates) can be chlorinated without encountering these difficulties. An anhydrous chlorinating agent is used containing ⩽ the same wt. of $AlCl_3$ as anthraquinone compound and ⩽ 1 mol. of SO_3 or R-SO_2-Hal (R = OH or O-Metal) or a non-aromatic organic radical/mol. of anthraquinone. The probable course of reaction is—

(1) $RSO_2Cl + AlCl_3 \rightarrow [AlCl_3RSO_2]^- Cl^+$

(2) $[AlCl_3RSO_2]^- Cl^+ + X{-}H \rightarrow [AlCl_3RSO_2]^- H^+ + X{-}Cl$

(X–H = cpd. to be halogenated). Reaction is carried out, preferably, at 85–135°c. in 1–3 hr. Thus, anhydrous $AlCl_3$ (80), NaCl (13) and NaF (0·8) are heated to a homogeneous melt at 150°c. After cooling to 90°c., $ClSO_3H$ (5) is added dropwise. Dibenzanthrone (5) is then added at 87–90°c. and the mixture stirred at 87–90°c. for 1 hr. The melt is poured on to ice and water, acidified to Congo with HCl, heated to 90°c., filtered, washed neutral and dried giving a product (6·1) containing 20% Cl. 22 other examples are given. E.T.

Fluorescent Brightening Agents
American Cyanamid Co. *USP* 2,844,539

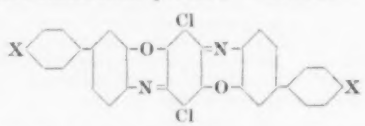
Addition of maleic, fumaric, oxalic, tartaric or benzene phosphonium acid to a fluorescent brightening agent of the type of strong inorganic salts of coumarin, e.g. 4-methyl-7-diethylaminocoumarin sulphate, yields a composition having good solubility in water. An aqueous solution of such a composition shows no hydrolysis or precipitation of the coumarin compound when it is diluted. C.O.C.

Readily Dispersible Dye Powders
FH *BP* 807,089

The products obtained from an aqueous suspension of a dye, especially a vat dye or its leuco acid, there also being in the suspension a dispersing or other auxiliary agent if desired, by drying it while it is frozen, are powders which are readily dispersed in water. C.O.C.

Rendering Dioxazine Pigments Resistant to Flocculation
DuP *USP* 2,843,498

The reddish violet pigment 6:13-dichloro-2:9-diphenyltriphendioxazine, disclosed in *BP* 646,099 (J.S.D.C., **67**, 124 (1951)) which has good fastness to light and very high tinctorial value has found little commercial use because of its very severe flocculation in paint systems. This drawback is overcome by treating the pigment or its dibromo- or dichloro-substitution products of formula—



with 3·5–25·0% of the aluminium salt of a mono- or dicarboxy-6:13-dichloro-2:9-diphenyltriphendioxazine or a 6:13-dichloro-2:9-diphenyltriphendioxazine polysulphonic acid. The treated pigments are similar tinctorially to the untreated pigments but show excellent resistance to flocculation in various paint systems. They are obtained by slurrying the pigment with the required amount of sodium dioxazine carboxylate or sulphonate and precipitating with aluminium sulphate. C.O.C.

Salts of Sulphuric Acid Esters of Leuco Anthraquinone Vat Dyes

DH *BP* 806,634

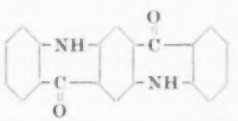
The present patent is a further extension of the work on sulphuric acid esters of *leuco* anthraquinone vat dyes which are difficult to esterify. Earlier methods are the subjects of *BP* 719,621; 745,365; 745,368 and 766,638 (J.S.D.C., **71**, 157 (1955); **72**, 394 (1956); **73**, 226 (1957)). Vat dyes difficult to esterify may be determined by a test given in *BP* 719,621. In the present method the vat dye is treated in a mixture of diethyl*cyclo*hexylamine and/or ≤ 1 diethyl-(mono-methyl-*cyclo*hexyl)-amine and ≤ 1 inert liquid diluent having a dispersing action on chlorosulphonic acid addition products of the amines. Such mixtures contain 30–90% of the amine(s); finely divided (150–200 mesh) metals, e.g. Fe, Co, Zn, Cu, are used and these may be activated as described in *BP* 766,638. Chlorosulphonic acid (40) is added dropwise to a cooled, stirred mixture of 1:2-dichloroethane (80), diethyl*cyclo*hexylamine (120) and brass powder (10) and 1:5-dibenzoylaminoanthraquinone (10) are added. The melt is stirred 5 hr. at 58–60°C., poured into 8% aq. Na_2CO_3 solution (1000) and the 1:2-dichloroethane and diethyl*cyclo*hexylamine removed by vacuum distillation over a heating bath at 50°C. The precipitate is filtered, heated 30 min. at 35–40°C. with alcohol (100), and 30% NaOH (200) in water (250). Filtration removes metal residues and, after concentrating the filtrate under vacuum (50°C.) the sulphuric acid ester salt of *leuco*-1:5-dibenzylaminoanthraquinone is salted with NaCl (90% yield).

E.T.

α-Linear Quinacridone— Red Pigment

DuP *USP* 2,844,484

Linear quinacridone—



is produced in the *α* form in small particle size without any undesirable phase or colour change occurring by milling any crystal phase of quinacridone with a relatively large amount of salt until a sample of the pigment separated from the salt has surface area > 60 sq.m./g. when measured by Emmet's nitrogen absorption method (*Advances in Colloid Science*, Vol. 1 (1942)). The salt is then extracted with dilute aqueous acid to leave the quinacridone in a pure *α* form. It is intensely red and of small particle size. C.O.C.

β-Linear Quinacridine— Violet Pigment

DuP *USP* 2,844,485

Quinacridone, in any crystal phase, when milled with a relatively large amount of salt in presence of xylene until a sample of the pigment separated from the salt, has surface area > 60 sq.m./g. when tested by Emmet's nitrogen absorption method, yields after separation of the salt and xylene, pure *β*-form quinacridone which is an intense violet pigment of small particle size. C.O.C.

γ-Linear Quinacridone— Red Pigment

DuP *USP* 2,844,581

The product obtained by salt milling any crystal phase of quinacridone, wetting the milled mixture with dimethylformamide and then extracting the salt and the dimethylformamide with dilute aqueous acid is an intense red pigment and consists of quinacridone in the *γ*-crystal phase. C.O.C

Organophilic Hydrophobic Silicates, Silicas, Silicic Acid, and Hydrated Silicas— Fillers for Rubber and Plastics and Vulcanisers for Silicone Rubber

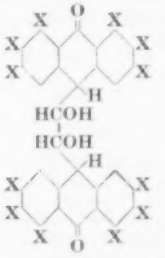
General Electric Co. *BP* 806,604

Hydrophilic silicates, silicic acid, silicas, and hydrated silicas are rendered organophilic and hydrophobic by esterifying their surfaces. This is effected by treating the finely powdered silica etc. with ethanol in presence of a hydroxide of an alkali metal or alkaline earth metal. The products are valuable fillers for rubber and plastics. In addition they reinforce and effect vulcanisation in silicone rubber without use of the usual cross-linking agents. C.O.C.

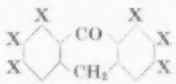
Anthrone Derivatives— Vat Dye Intermediates

ICI *BP* 806,384

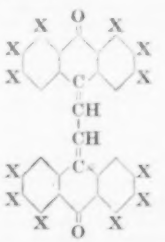
Compounds of formula—



(X = H or Hal) are intermediates for acedianthrone vat dyes. They are made by heating glyoxal and an anthrone of formula—



in a neutral water-miscible organic solvent, e.g. ethanol, acetone, acetonitrile. Reaction of these products with dehydrating agents yields—



(cf. *BP* 296,817) which may be converted to acedianthrones by the method of *BP* 551,622 (J.S.D.C., **59**, 155 (1943)). Anthrone (19·4), glyoxal polymer containing 78% glyoxal (3·9) and 90·5% ethanol (100) are stirred 5 hr. under reflux. White, crystalline 1:2-di-(anthr-9-on-10-yl-)ethane-1:2-diol is formed and filtered off and dried (m.p. 230°C.). This product (11·2) is added to nitrobenzene (120) and acetic anhydride (32·5), stirred at 70°C. and conc. H_2SO_4 (0·5) added over 15 min. After a further 15 min. the mass is cooled to 15°C.; the 1:2-di-(anthr-9-on-10-ylidine-)ethane is filtered off and the cake is freed from nitrobenzene by steam-distillation before final filtration. Alternatively, the acedianthrone may be made without isolating the above intermediate. Thus, the above nitrobenzene melt is stirred at 15–20°C., conc. H_2SO_4 (16·2) is added and the melt is heated to 50°C. in 2 hr., at 50°C. for 1 hr.; then to 95°C. in 1 hr. and at 95°C. for 1 hr. Cooling, filtration, washing with nitrobenzene and solvent removal by steam follow. E.T.

Caramel (C.I. Natural Brown 10)

Baker Perkins *BP* 806,633

Apparatus for effecting continuous caramelisation. The length of time of caramelisation can be varied without interfering with the rate of flow of the material through the apparatus. C.O.C.

Pelleting of Metal Silicates

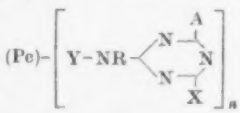
Godfrey L. Cabot *USP* 2,844,444

The metal silicates, and other normally non-agglomerative powders, e.g. whiting (C.I. Pigment White 18) are rendered agglomerative by grinding them to average particle size $< 2{\cdot}0\ \mu$., wetting them, forming them into a dry cake and finally breaking the cake into agglomerates of irregular shape and particle size $> 2{\cdot}0\ \mu$. The resulting pigment is readily converted into pellets by simply agitating it in a revolving drum. The process is particularly applicable to the deposit of wollastonite found near Willsboro, New York, which is brilliantly white (92–96% standard reflectance) and of high chemical purity (> 98% $CaSiO_3$). C.O.C.

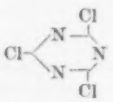
Phthalocyanines Containing Halogenocyanuryl Radicals

ICI *BP* 805,562

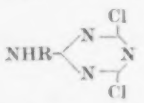
Blue to green dyes for cotton, wool, silk, polyamide or polyurethane fibres are of formula—



(Pc = phthalocyanine radical; Y = direct link or divalent bridging radical; (Pc)–Y– contains < 1 solubilising group; R = H, Alk, aralkyl, *cyclo*alk; X = Hal; A = Hal or O–B where B is a subst. or unsubst. hydrocarbon; n = a whole no.). 25 examples of the bridging radical Y are given including $-SO_2$–phenylene–, –CONR–arylene– etc. Examples of solubilising groups are $-SO_3H$ and –COOH and the phthalocyanine nucleus may be metal-containing or metal-free and may contain other substituent groups, e.g. Cl, Br, aryl. Preparation is by (1) reacting an aminophthalocyanine (Pc)–(Y–NHR)$_n$ with a triazine, e.g.



or (2) when Y stands for $-SO_2$– or –CO–, reacting (Pc)–$(SO_2Cl)_n$ or (Pc)–$(COCl)_n$ with an aminotriazine—



For example, starting from Cu phthalocyanine (288), the tetrasulphonchloride is prepared by the method of *BP* 515,637; the resulting moist cake is suspended in ice-water (3000), $NaHCO_3$ (336) is added followed by solutions of 2:5-diaminobenzene sulphonic acid (188) in water (1500) and N. $NaHCO_3$ (1000). After 18 hr. stirring, the temperature rising to 25°c., 38% HCl (360) is added, the mixture is filtered and the residue washed with 2 N. HCl. Drying gives the blue intermediate Cu phthalocyanine-*N*-(4-amino-3-sulphophenyl-)sulphonamide-sulphonic acid. This product (56·8) is dissolved in water (3000) and 2 N. Na_2CO_3 solution to give pH 7. The solution is added dropwise during 30 min. to a suspension of cyanuric chloride (20) which has been prepared by dissolving the cyanuric chloride in acetone (80) and pouring into ice-water (300) at 0–10°c. After 3 hr. stirring the mixture is filtered; the filtrate is adjusted to pH 7 with 2 N. Na_2CO_3 and diluted to 5000. A solution of disodium hydrogen phosphate (60) and potassium dihydrogen phosphate (120) in water (1000) is added followed by NaCl (1000). After filtering, the precipitated dye it is washed with a solution of NaCl (500), Na_2HPO_4 (30) and KH_2PO_4 (60) in water (2400). The finished dye is applied to cotton by padding and aftertreatment with aq. NaOH and gives greenish-blue dyeings fast to washing. 15 other examples are given. E.T.

Pelleting Carbon Black (C.I. Pigment Black 6 and 7)

Phillips Petroleum Co. *USP* 2,844,445

By spraying water on to the pellet recycle steam from a pellet mill so as to wet the pellet without forming carbon black mud in the conveyor there is reduction in the loose pigment in the effluent steam from the mill. In addition a denser pellet can be produced at standard production rate or the production rate can be increased when producing pellets of standard density. C.O.C.

Water-dispersible Talc (C.I. Pigment White 26)

Sierra Talc & Clay Co. *USP* 2,844,486

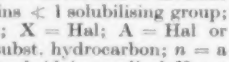
A water-dispersible product particularly suitable as a pigment for coating paper is obtained by disintegrating the talc in presence of 1–4% on the weight of the pigment of a non-ionic surfactant whose hydrophilic portion consists of ethylene oxide groups, e.g. Igepal CO (General Aniline). C.O.C.

Acylaminomethylene Phthalocyanine Dyes

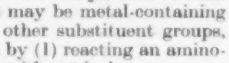
BASF *BP* 806,175

The products of this invention are either (*a*) water-insoluble pigments or (*b*) water-soluble dyes for fibrous materials of all kinds, natural and synthetic resins, lacquers or plastics; the latter are acid salts produced by acid hydrolysis of the former. They are of formulae—

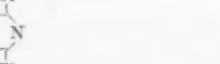
((Pc) = metal-containing or metal-free phthalocyanine radical; R^1, R^2 = H or Alk; Z = H, Hal, alkoxy, dialkylamino, alkylthio; n = 2–8) or—



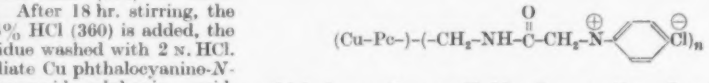
(Y = quaternary ammonium, tertiary sulphonium or isothiouronium group). They are made by reacting a phthalocyanine in a liquid or molten medium of an anhydrous acid or anhydrous Al halide with a compound—



(A = OH, alkoxy, acyloxy, Hal) or, by reacting a phthalocyanine with a mixture comprising—



and a monomeric or polymeric HCHO or dichlorodimethylether. Thus, P_2O_5 (50), Cu phthalocyanine (60) chloracetamide (110) and paraformaldehyde (60) are added to 100% H_2SO_4 (800) whilst stirring and cooling. After heating to 100°c. for 20 hr., the cooled mixture is added to ice water. Filtration, washing and drying gives (Cu–Pc)–$(CH_2{\cdot}NH{\cdot}CO{\cdot}CH_2Cl)_n$ (85–90). This product (30) is ground and stirred with pyridine (300) in a water-bath for 1 hr. After cooling, the dye is precipitated with acetone, filtered and dried at low temperature giving—

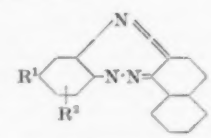


which dyes cotton or silk blue from acetic or formic acid baths. E.T.

Fluorescent Brightening Agents for Nylon and Organic Derivatives of Cellulose

General Aniline *USP* 2,842,501

Compounds of formula—



(R^1 = amino, *NN*-dialkylamino, *NN*-bis-β-cyanoethylamino or *N*-morpholino; R^2 = H, Alk, alkoxy or Hal), e.g. 4-[naphtho(1,2)triazolyl(2)]aniline, are fluorescent brightening agents for nylon, acetate rayon, etc. to which they are applicable from detergent solutions. C.O.C.

Titanium Dioxide (C.I. Pigment White 27)

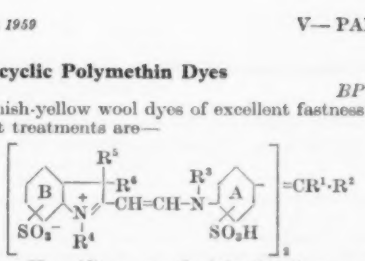
American Cyanamid Co. *USP* 2,842,428

Rutile TiO_2 free from phosphatic, iron, vanadium, and chromium impurities is obtained by treating a washed titanic hydrolysate containing such impurities with caustic, forming an insoluble metal titanate, solubilising the alkali-soluble impurities and washing them out, and calcining at a temperature which completes the reaction of the caustic but does not cause formation of anatase. The metal titanate is then digested with conc. H_2SO_4 and the acid-soluble impurities solubilised. A solution of titanium sulphate is formed from the cake, filtered, hydrolysed, the precipitate washed free of acid-soluble impurities and calcined to form the pigment. The product has outstanding brilliancy, tinting strength, and oil absorption. C.O.C.

Heterocyclic Polymethin Dyes

ICI *BP* 806,817

Greenish-yellow wool dyes of excellent fastness to light and wet treatments are—



(R^1, R^2 = H or Alk, or may be joined to form a *cyclo*alkyl ring which may contain alkyl substituents; R^3 = Alk or alkylene connected to ring A *ortho* to the N atom to form a heterocyclic ring; R^4, R^5, R^6 = same or different Alk; rings A, B may be further substituted). The unsulphonated intermediate products for these dyes are made by reacting a *cyclo*alkanone or an aliphatic aldehyde or ketone with $\leqslant$ 2 mol. of an *N*-alkylaniline, an indoline or a tetrahydroquinoline at elevated temperature in a medium containing $\leqslant$ 1 equiv. of an acid/mol. of amine. The wool dyes are then obtained by sulphonating. Thus, 2-methylindoline (266) and 36% HCl aq. (100) are stirred at 80–90°c. and 37% aq. HCHO (81) is added. After 16 hr. under reflux, the mixture is poured into water (2000) and Na_2CO_3 (106) and unreacted 2-methylindoline removed by steam. The residue, decanted from water, is distilled (b.p. 250–60°c. at 0·1 mm.) and is bis-(2-methyl-5-indolinyl-)methane. This product (278) in 30% H_2SO_4 (2000) is added at 20°c. to a solution of 2-aldehydomethylene-1:3:3-trimethylindoline (603) in 30% H_2SO_4 (2000). After stirring 48 hr. at 20°c., the resulting bis-[2-methyl-1-β-(1′:3′:3′-trimethyl-2′-indoleniniumyl-)-vinyl-5-indolinyl-]methane sulphate is filtered, washed with water (5000) and dried. The product (5) is added to 20% oleum (25) below 20°c. and stirred until solubility is attained. The melt is poured on to ice (50), cooled to 0°c., filtered and the solids re-crystallised from 17% NaCl and dried. E.T.

Bright Canary Yellow Phosphorus Sulphides

Monsanto *USP* 2,844,442

Phosphorus and sulphur are continuously introduced into a heel of boiling phosphorus sulphide in a combined reactor and still wherein the exothermic heat of the phosphorus–sulphur reaction effects continuous distillation of the reaction product. At the same time boric oxide is continuously introduced into the heel of boiling phosphorus sulphide to improve the colour of the product. The distillate is conveyed continuously to a hot condenser where the whole of the phosphorus sulphide vapour condenses while uncondensed gases and phosphorus sulphide gas are vented to the atmosphere. The resulting liquid phosphorus sulphide is further cooled and fed continuously into a chill roll which solidifies it and breaks it up into flakes. The flakes are then ground to powder. Alternatively the product from the reactor and still, without treatment with boric acid, is condensed and then distilled with simultaneous treatment with boric acid to give the desired bright yellow product. The product can be used *inter alia* as an anticorrosive agent and antioxidant for oils, extreme pressure lubricants, match heads and in vulcanised rubber, all of which it colours bright canary yellow. C.O.C.

Vanadium–Zirconium Pigments

Harshaw Chemical Co. *BP* 806,480

Yellow to reddish yellow vanadium–zirconium ceramic pigments are obtained by incorporating $\geqslant$ 5% of indium or gallium oxides, e.g. a mixture of ZrO_2 (94), NH_4NO_3 (5) and In_2O_3 (1) calcined at 1100°c. in a slightly reducing atmosphere yields a deep reddish yellow glaze stain. C.O.C.

Zinc Selenide

Philips Electrical Industries *BP* 806,566

Very pure zinc selenide is obtained by reducing zinc selenite with hydrogen at 900–1200°c. Preferably the zinc selenite is strewn or sprayed into a stream of hydrogen at the required temperature. There is a 70% yield compared with the 30% yield obtained when reduction is effected at 300–400°c. C.O.C.

Corrosion of Apparatus by Food Dyes (I p. 159)
1958 Reviewed— Technical Developments in the Paint and Printing Ink Industries (V this page)
Zinc Oxide Paints (V below)
Rôle of Pigments in Weathering of Automotive Paints (V below)
Substituted Benzidines and Related Compounds as Reagents in Analytical Chemistry. XV— Some New 3,3′-Disubstituted Naphthidines (XIV p. 179)
Colorimetric Determination of Rutin (C.I. 75730) (XIV p. 180)

V— PAINTS; ENAMELS; INKS

1958 Reviewed— Technical Developments in the Paint and Printing Ink Industries

W. G. B. Mills

Paint, Oil & Colour J., **135**, 97–102 (9 Jan. 1959)

A listing of the literature indicating in each case the ground covered by the author. It is divided into the following sections— Pigments (26 references), Paints (20), Varnishes (7), Oils (11), Resins (28), Plant and Equipment (6), Analysis and Testing (6), Printing Inks (6), and Corrosion and Weathering (9). C.O.C.

Zinc Oxide Paints

P. Condi

Chim. e Ind. (Milan), **40**, 345–355 (1958): *Chem. Abs.*, **52**, 21152 (25 Nov. 1958)

Analysis of the effect of particle size and shape of ZnO (C.I. Pigment White 4) on the properties of linseed oil paint showed that: oil absorption is inversely proportional to the apparent d.; the latter is appreciably smaller for acicular ZnO than for the modular variety. The viscosity (measured in a 1:1 suspension by a rotary Wolf–Hoepke viscometer) is a function of the "sediment volume" (apparent volume : real volume) of the pigment and follows Robinson's equation (*Chem. Abs.*, **44**, 904 (1950)). Hiding power increases with decreasing average particle size of ZnO, the optimum being 0·4 μ.; pigments with narrow scatter in particle size are better than those having wide deviation from the average size. C.O.C.

Rôle of Pigments in Weathering of Automotive Paints

S. B. Twiss, W. L. Weeks, and F. O. Thomas

Am. Soc. Testing Materials Proc., **57**, 1337–1366 (1957): *Chem. Abs.*, **52**, 21154 (25 Nov. 1958)

Optical and electron microscopical study of the surface structure of paint films weathered for 1 year in Florida shows that with pigments of $>$ 100 mμ. diameter the erosion and shrinkage of the binder releases discrete particles on the surface in the form of chalk and the gloss falls rapidly. Such pigments are TiO_2 (C.I. Pigment White 4), Red Ferric Oxide (C.I. Pigment Red 101), Ferrite Yellow (C.I. Pigment Yellow 42), Lamp Black (C.I. Pigment Black 6) and Copper Maroon (C.I. Pigment Brown 9). With pigments $<$ 100 mμ. diameter, detached thin sheets of pigment binder lie on the surface and produce bronzing; this class includes Iron Blue (C.I. Pigment Blue 27), Gold Paste (C.I. Pigment Yellow 42), Thio Fast Red (C.I. Pigment Red 88), Carbon Black (C.I. Pigment Black 7), Burnt Sienna (C.I. Pigment Brown 7), Chrome Green (C.I. Pigment Green 15), Amberine Maroon, Maroon Gold and Phthalocyanine Blue (C.I. Pigment Blue 15). Pigments having a dual size range, including Chrome Yellow (C.I. Pigment Yellow 34), Chrome Orange (C.I. Pigment Orange 21), Molybdate Orange (C.I. Pigment Orange 104) and Toluidine Red (C.I. Pigment Red 3) show both chalking and bronzing. Checking is a sign of degradation deep in the paint film; it indicates that the pigment does not protect the underlying binder from damaging radiation. Checking occurs in unpigmented films and with Caddy Red, Dinitroaniline Orange, Phthalocyanine Green (C.I. Pigment Green 7), Green Gold (C.I. Pigment Green 10) Indo Blue (C.I. Pigment Blue 21), Indo Orange, Chrome Hydrate Green, and BON Red. Chalking or bronzing usually accompanies checking. C.O.C.

Lac in Surface Coatings

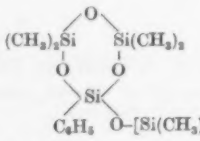
S. P. Potnis and M. G. K. Rao

Paintindia, **7** (11), 26–34 (1958): *Chem. Abs.*, **52**, 21158 (25 Nov. 1958)

Review of the composition of shellac and its use in protective coatings. 61 references. C.O.C.

Effects of Pigments and Catalysts on the Heat-ageing of Poly(methylphenylsiloxane) Coatings
K. A. Andrianov and M. B. Fromberg
Khim. Prom., 12–17 (1958): *Chem. Abs.*, **52**, 21154 (25 Nov. 1958)

The thermal oxidative destruction of poly(methylphenyl siloxane)—



which contains alternating spacial and linear links, proceeds with a break-off of organic radicals on the Si–C band and the destruction of the Si–O chain band. Catalysts used to accelerate drying of the siloxane film intensify the destructive and the thermal ageing processes. The mechanism of the destruction of the siloxane varied with different catalysts. Salts of metals having constant valency favour more intensive decomposition of the siloxane at the Si–O band only. Those of metals having variable valency acted catalytically in the rupture of siloxane bonds and the oxidation of the radicals. Pigments and fillers added to the siloxane reduced the thermal elasticity of the films, while the thermal stability of polyester-modified siloxane was greatly increased by pigmentation. Loss in weight during thermal oxidation of the films was reduced by most pigments and the stability of the film at higher temperatures was greatly increased. C.O.C.

Organic Finishing of Aluminium and its Alloys
L. F. Spencer
Metal Finishing, **56** (8), 58–61 (1958): *Chem. Abs.*, **52**, 21157 (25 Nov. 1958)

A general account of the use of organic finishes on aluminium and its alloys, including surface preparation, primers and final finishes. These include synthetic resin formulations (styrenes, polyamides, acrylates, ethylcellulose, vinyls, alkyls, melamines, silicones, phenolics, ureas, urea-formaldehyde, furans), bituminous paints and luminous paints. C.O.C.

Improving Black Printing Inks
B. I. Berezin
Poligraf. Proizvodstvo, (3), 26–7 (1955): *Referat. Zhur., Khim.*, Abstr. No. 48880 (1956): *Chem. Abs.*, **52**, 21155 (25 Nov. 1958)

Improvement in quality of black printing inks (for rotary and flat machines) is achieved by increasing the amount of oleic acid in the "induline melt" from 1 to 1·25 parts of oleic acid per part of induline. This increases the stability of the dye, permits increase in the amount of Carbon Black (C.I. Pigment Black 6 and 7) used, decrease in the amount of dye needed, and improves the degree of fixation and printing properties. The stability of the dye in black inks for printing illustrations with a flat machine (chiefly for use on high gloss paper) was improved by use of a synthetic alkyd drying oil instead of the linseed oil previously used, it also gave quicker drying and better printing properties. C.O.C.

PATENTS

Thixotropic Aqueous Coating Compositions
DuP *BP* 806,556

A thixotropic coating composition comprises an aqueous dispersion of polyvinyl (acetate), a styrene–butadiene copolymer or an acidic copolymer of methyl methacrylate and an acrylic ester of a saturated aliphatic monohydric alcohol of 1–4 C pigment and 0·3%–2·0%, on weight of the composition, of finely divided, water-swellable hydrous silicate. The composition must have "plastic viscosity" of $\lesssim$ 0·6 poise at 25°c. and a "hysteretic viscosity loss" of $\lesssim$ 20 units measured at 140 r.p.m. with a Precision—Interchemical Rotational Viscosimeter at 25°c. C.O.C.

Can-stable Coating Composition
ICI *BP* 807,199

A composition which may be stored without permanent gelation occurring consists of the reaction product of a resin contaniing free hydroxyl groups and an unsaturated urethane *iso*cyanate in a non-reactive solvent. C.O.C.

Heat-resistant Enamelling Paint containing Silicone Resin
U.S. Secretary of the Army *USP* 2,843,559

A paint for application to ferrous metal articles to provide good rust-protection at temperatures up to 1600°F. is produced by incorporating ceramic or vitreous frits as part of the pigment content of a conventional paint. At normal temperatures the frits act as pigments in the paint film, but when heated, as the fusion point of each frit component is reached, each component progressively melts and fuses to the base metal to provide a ceramic type of vitreous film resembling an enamel coating. The fused frit replaces the organic or semi-organic vehicle or binder which is destroyed by the heating. C.O.C.

Aluminium Stoving Enamels which retain their Leafing Value on Storage
DuP *USP* 2,843,554

An enamel which retains its leafing value both during storage at room temperature and when vigorously agitated or stirred when aerated as in dip coating, is obtained by mixing an extra fine particle size leafing aluminium pigment (C.I. Pigment Metal 1) with a film-forming oleoresinous esterification product of (1) rosin acids, (2) a mixture of fatty acids comprising mainly polyetheroid acids, (3) an esterifiable epoxyhydroxy polyether resin obtained by condensing epichlorohydrin or glycerol dichlorohydrin with a bis-(4-hydroxyphenyl)-alkane, and a volatile hydrocarbon solvent. The enamel is applied and the coated article heated to $\lesssim$ 300°F. to cure the coating. C.O.C.

Thermo-fluid Vehicles for Application of Vitreous Enamel Colours
DuP *USP* 2,842,454

A vehicle which readily melts to a liquid at 120–212°F., does not run while molten, levels off well before solidifying and which solidifies rapidly at room temperature consists of natural vegetable wax (3·5–47·5%), paraffin wax (17·5–85·5), aluminium stearate (2·0–20·0), phosphorated tall oil (0–3·0%), and stearic acid and/or stearyl alcohol (0–68·0%). C.O.C.

Liquid Bright Gold—Precious Metal Decorating Compositions
DuP *USP* 2,842,457

A precious metal resinate, a flux for the metal, and thermosetting epoxy resin are dissolved in a suitable solvent to yield a solution which is readily applied to a ceramic and set up to a hard surface which on being fired yields a strongly adherent, brilliant metallic finish. C.O.C.

Carboxymethylcellulose Acids and Complex Ammonium Zirconium Carbonates for forming Water- and Alkali-resistant Coatings
Sherwin-Williams Co. *USP* 2,842,451

An aqueous solution containing carboxymethylcellulose acids and a complex ammonium zirconium carbonate, e.g. $(NH_4)_3HZrO(CO_2)_3$, are used at pH 7·5–9·0 to produce coatings which when dried are resistant to water, soap solutions, etc. C.O.C.

Polymethyl Siloxane Heat-resistant Ink
Lockheed Aircraft Corpn. *USP* 2,844,550

Inks made up of a silicone resin of good thermal stability, e.g. a polymeric silicone oxide having a CH_3:Si ratio of 1 : 2, pigment, carrier or thinner, dispersing agent and, preferably, a drier are resistant to heat up to 1000°F. They do not corrode metal. C.O.C.

Metal-complex Disazo Dyes—Water-soluble Blacks for Inks (IV p. 165)

VI—FIBRES; YARNS; FABRICS

Chemical Modification of Cotton—Progress and Current Status
C. H. Fisher and F. S. Perkerson
Text. Research J., **28**, 769–778 (Sept. 1958)

The authors list the more important modifications of cotton in order of their use in industry from around 1907. They then discuss in more detail the advances made since 1950. The low cost of raw materials (cotton and many chemicals) make the modified cottons attractive when competing against the more expensive man-made fibres. An extensive list of references is given. S.B.D.

Deformation of Networks by Detachment and Formation of Cross-links
F. Oosawa
J. Polymer Sci., **32**, 229–246 (Oct. 1958)

Cyanoethylation of Cotton in Aqueous Medium
N. M. Bikales and L. Rapoport
Text. Research J., **28**, 737–743 (Sept. 1958)

An investigation was made of the cyanoethylation of cotton using small quantities of acrylonitrile dissolved or dispersed in sodium hydroxide soln. As compared with conventional processes, both the rate of reaction and the max. degree of cyanoethylation were lower, although very little acrylonitrile was lost in side-reactions. A study of the physical properties of the product showed the importance of reaction conditions in determining the properties of cyanoethylated cotton. S.B.D.

Yellow Spot Disease of Cotton
M. Nopitsch, E. Möbus, and D. Havenith
Melliand Textilber., **39**, 1268–1271 (Nov. 1958)

Living or stored cotton suffering from visible yellow spot fluoresces a bright greenish yellow in ultraviolet radiation, unlike cotton affected by cavitoma, and is not alkaline like cavitoma cotton. Nutrient media are described which allowed isolation from diseased cotton and identification of 2 bacteria—*cellvibrio vulgaris* yellows cotton but only fluoresces slightly—and 7 mildews, listed in decreasing order of frequency. *Aspergillus flavus* is mainly responsible for the disease. It was not found on cavitoma cotton, from which 7 fungi were isolated, which are also listed. Marsh's findings (*Text. Res. J.*, **25**, 1007–1016 (1955)) that *Aspergillus flavus* infections could only start in the living plant were substantiated. Infection spreads only with R.H. $> 70\%$, acid conditions favouring moulds and neutral or weakly alkaline conditions bacteria, which require even higher humidities. 9 photomicrographs illustrate the splits distinguishing diseased from smooth, healthy cotton swollen in 19°Bé NaOH, and some of the cellulose-destroying organisms. S.M.J.

Fine Structure Determination of Cellulosic Fibres
T. Koshiro, K. Yagami, E. Shiratsuchi, and T. Fujimura
Bull. Chem. Soc. Japan, **31**, 606–611 (July 1958)

Simultaneous oxidation and hydrolysis of cellulose with $K_2Cr_2O_7$ and H_2SO_4 appears to remove the amorphous regions, leaving the original crystalline regions unchanged, although the resulting high alkali solubility suggests the introduction of carboxyl groups. A.J.

Viscose Rayon—Stabilisation by Heat Treatment
K. Kanamaru, N. Tokita, and S. Noyama
J. Polymer Sci., **31**, 359–382 (Sept. 1958)

Viscose rayon was swollen in very dilute H_2SO_4 (pH 2·4–5·1), and heat-treated at 60–200°c. The following properties were studied after the various treatments—transition modulus of elasticity and other constants in a three-parameter viscoelasticity model, dynamic modulus of elasticity, breaking strength and elongation at break, crystallinity, water absorption. Heat treatment after fibres are swollen at pH 3·0–5·0 and at 60–70°c. (close to the glass transition temperature of cellulose hydrate) was the most effective. From the results it is concluded that heat treatment mainly affects the amorphous region of the viscose rayon, creating new ordered regions. P.T.S.

Metallic Threads
E. Frieser
Z. ges. Textilind., **60**, 757–759 (5 Sept. 1958)

The new aluminium-based metallic threads are described with special reference to Lurex. The properties of the various types of Lurex thread are summarised, and points relevant to the finishing of fabrics containing Lurex are noted. There are brief notes on the following types of metallic thread—Metlon, Cellometall, Conomet, Fairtex, Lame, and Malora. J.C.F.

Jute—Decrease in Lateral Order when treated with Caustic Soda Solutions
V. D. Gupta
J. Polymer Sci., **31**, 219–221 (Aug. 1958)

An X-ray method shows a slow change in lateral order with dilute solutions. With solutions between 10 and 20% (i.e. near mercerising strength) the change is rapid. P.T.S.

Fibres from Amylose Triacetate
R. L. Whistler and G. N. Richards
Ind. Eng. Chem., **50**, 1551 (Oct. 1958)

Fibres of amylose triacetate were spun from solutions in chloroform and methylene chloride. Undrawn fibres had lower tenacity (0·55 g./denier) but much greater elongation at break (ca. 60%) than cellulose triacetate. Drawn fibres had somewhat higher tenacity, but in general had no advantage over cellulose triacetate. W.K.R.

Corn Amylose and Amylose Triacetate Fibres
I. A. Wolff
Ind. Eng. Chem., **50**, 1552 (Oct. 1958)

Fibres prepared from amylose triacetate are improved in both wet and dry strength by orientation produced by stretching under suitable conditions, up to 1·8 g./denier/dry being attained with 600% stretch. Amylopectin triacetate fibres had low tenacity (0·23 g./denier) and were brittle. Amylose fibres (undrawn), prepared by saponification of the triacetate fibres, also had low tenacity (0·29 g./denier). W.K.R.

Structural Compactness of Woven Wool Fabrics and their Behaviour in Modern Washing Machines
H. Bogaty, G. H. Lourigan, and H. E. Harris
Text. Research J., **28**, 733–737 (Sept. 1958)

Many all-wool fabrics of varied construction were given 10 consecutive mild washes in a domestic washing machine, and the resulting shrinkage was determined. The shrinkage of fabrics of the same weave type was shown to be related to the cover factor of the cloth. A measure of compactness of fabric structure could be obtained from the ratio of the actual to the max. weavable cover factor. This compactness ratio could be related to shrinkage in laundering for nearly all fabrics, irrespective of weave. This relationship indicated that a commercially acceptable fabric could be designed with respect to its compactness to give low levels of felting in repeated washes. S.B.D.

Felting of Wool
R. Walther
Melliand Textilber., **39**, 1240–1243 (Nov. 1958)

Known work is clearly reviewed from the point of view of the relative influence of geometrical and physical properties of wool fibres and of processing variables in felting. 20 references are provided. S.M.J.

Degradation of Cystine in Alkaline Solution
R. L. Elliott, R. S. Asquith, and M. A. Hobson
J.S.D.C., **74**, 859–860 (Dec. 1958)

Rabbit Hairs

XVIII—Frictional Properties of Angora Rabbit Hairs in Water
S. Ikeda, S. Okajima, and T. Inoue
Sen-i Gekkaisha, **14**, 15–20 (1958)

Apparatus is described which uses the inclined-hair method with a Pt rider weighing 14·2 mg. and 0·3 mm. diameter. Frictional coefficients increased when the hairs had been degreased with CCl_4. The effect of the thickness of the fur on the frictional coefficient was negligible. For single hairs the apparatus can give a definite value.

XIX—Effect of Carrotting Conditions upon the Increase in Weight of the Carrotted Fur
S. Ikeda and S. Okajima
Ibid., 175–182:
Chem. Abs., **52**, 21190 (28 Nov. 1958)

Fur was treated at 20–60°c., for 5–85 min. with 0·5–4·5% $Hg(NO_3)_2$, 1·0–5·0 HNO_3, 0·1–0·5% $NaNO_2$. The experiments were designed by use of a 5×5 Hyper Graeco–Latin Square and L_{25} (3^{12}). The fur was carrotted, centrifuged dried at 95°c. for 30 min., washed and dried at 95°c. to constant weight. The weight increased linearly with the concentration of HNO_3 and $NaNO_2$ and the immersion time and quadratically as the temperature of the carrotting liquor increased. $Hg(NO_3)_2$ (0·5–4·5%) has little effect upon the increase in weight at the 5% level. C.O.C.

Heat effect of Unoriented Amorphous Polyethylene Terephthalate Fibre
S. Sakajiri
J. Polymer Sci., **32**, 252–253 (Oct. 1958)

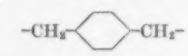
An X-ray diffraction pattern of unoriented polyethylene terephthalate fibre after heating for 15 min. at 150°c. under tension. P.T.S.

Polyethylene Terephthalate Birefringence— Effect of Steeping in Organic Liquids
S. Sakajiri
J. Polymer Sci., **31**, 198–199 (Aug. 1958)
Acetone, xylene, benzene, benzyl alcohol, and dioxan are not solvents or strong swelling agents for polyethylene terephthalate, but during 20 min. steeping they alter the birefringence of fibres considerably. P.T.S.

PATENTS

Improving the Dyeing Affinity of Polyvinyl Chloride Fibres
Montecatini Societa Generale per l'Industria Mineraria e Chimica *BP* 807,086
The affinity of polyvinyl chloride fibres for disperse, vat, and basic dyes is increased by incorporating in the spinning composition 1–15% of an acrylonitrile–butadiene copolymer. W.G.C.

Polyarylene-ethylenes
DuP *BP* 807,196
A process is described for the preparation of polymers having recurring—



units which are capable of being melt spun to form continuous orientable fibres, e.g. *p*-xylylene. W.G.C.

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Scouring of Wool-Terylene Cloth
R. S. Hartley and F. F. Elsworth
J. Textile Inst., **49**, P 554–P 564 (Oct. 1958)
The combing oils used at present in the worsted industry are less easily removed from Terylene than from wool, but they become easier to remove from Terylene as they are oxidised— in air and light, for example. Non-ionic surface-active agents added to the combing oil seem to be a more practical way of dealing with the problem than changing the scouring routine. P.T.S.

Detergent Adsorption at a Solution-Air Interface by Radiotracer Method. I— Adsorption Isotherms for Solutions of Sodium Alkyl Sulphates
R. Matuura, H. Kimizuka, S. Miyamoto, and R. Shimozawa
Bull. Chem. Soc. Japan, **31**, 532–538 (July 1958)
The surface adsorption of soln. of the labelled sodium salts of dodecyl, tetradecyl, and hexadecyl sulphates has been measured by means of a Geiger–Müller counter. Surface adsorption increases with increasing concn., reaching saturation near the critical micelle concn., and with increasing length of the hydrocarbon chain. Saturation concn. are near the theoretical values anticipated by the Gibbs equation. A.J.

Bleaching Cellulose with Sodium Chlorite
O. Deschler
Melliand Textilber., **39**, 1117–1120 (Oct. 1958)
The development of $NaClO_2$ bleaching is reviewed. It was initially applied to regenerated cellulose because of the low rate of attack on the substrate combined with rapid bleaching action. In bleaching linen yarn lignins are rapidly attacked, and data are given to substantiate the lower degree of damage associated with the sequence soda boil— acid NaClO–$NaClO_2$–peroxide contrasted with the slower alkaline NaClO as third process. Linen and linen–cotton fabrics are then discussed. Reasons for the later introduction of chlorite into cotton bleaching are enumerated. $NaClO_2$ instead of soda boil-NaClO produces stronger and whiter viscose rayon–cotton blends. In knitwear lower weight loss and fuller handle are obtained. Long- and short-liquor methods of bleaching cotton are reviewed, especially semi-continuous ones of the latter type, such as the pad–steam process. The additional corrosion hazard in this due to ClO_2 in the gas space can be countered by suitable activators providing acidity in the fabric and alkalinity in the gas space. Utility of higher fabric strengths in later finishing, lesser need for alkaline boiling except for dealing with dead cotton, and safe working are stressed. S.M.J.

PATENTS

Bleaching Composition
Olin Mathieson Chemical Corpn. *BP* 806,564
A combination of granular calcium hypochlorite and bead-form sodium tripolyphosphate can be used in heavy dosage in presence of soap without formation of calcium soap curds or "soap specks". It is readily soluble in warm water. C.O.C.

Continuous Bleaching of Cellulosic Textiles
ICI *BP* 806,536
Modification of *BP* 803,796 (J.S.D.C., **75**, 74 (Jan. 1959)). Use of acid aqueous hypochlorite instead of alkaline hypochlorite solution yields at least as good absorbency, colour and degree of mote removal and has the advantages of needing less careful control to avoid degradation of the cellulose and much less stringent precautions to avoid possible damage to the cloth in the hypochlorite bath if the plant is accidentally stopped. C.O.C.

VIII— DYEING

Influence of Fibre Types on Dyeing Methods
J. S. Ward
J.S.D.C., **74**, 813–823 (Dec. 1958)
An account is given of the many factors which influence the dyeing method adopted for a particular fibrous material. Some of these factors, such as the diffusion rate and the affinity of dyes, are fundamental; others, such as the colours required or variability in dyeing properties, are accidental; whilst many, such as the volume of business or the cost of dyes, are dependent upon the economic structure of the industry. All must be accommodated in a successful dyeing process.
AUTHOR

Individuality of Direct Dyes
R. A. Brooks
Dyer, **120**, 161–164 (1 Aug. 1958)
A review of factors influencing the substantivity and user properties of direct dyes. A.J.

Interactions Between Colorant and Hydroxylated Polymers
G. Centola
Ann. sci. textiles belges, **3**, 157–184 (1955): *Chem. Abs.*, **52**, 21121 (25 Nov. 1958)
Solutions of methylcellulose (1·58 OH per C_6) and poly(vinyl alcohol) were coloured with the direct dyes C.I. 24875, C.I. 23500, C.I. 24000, C.I. 24120, C.I. 24335, and C.I. 24260. The light absorptions of 15–20 g./l. solutions of methylcellulose and/or Na_2SO_4 were measured spectrophotometrically with addition of 0·01 millimole dye (base)/l. Addition of methylcellulose to dye solutions decreases the minimum transmittance and displaces the curves to a higher λ; Na_2SO_4 has the opposite effect. Addition of both methylcellulose and Na_2SO_4 displaces the curves as methylcellulose alone does. Scheibe's hypothesis (*Chem. Abs.*, **32**, 2835 (1938)) on the reversible association of dye molecules may explain these effects. The absorption spectrum varies with the degree of dye aggregation which increases with concentration, electrolytes present and decrease in temperature. Displacement of the curves varied with the degree of aggregation. Work using poly(vinyl alcohol) confirmed the hypothesis that the equilibrium between individual dye molecule and aggregates is influenced by electrolytes and hydroxylated polymers. X-Ray studies of films of methylcellulose confirm that the dye molecules penetrate those of the cellulose and reduce its crystallinity. All the dyes markedly increased the specific viscosity of solutions of methyl cellulose. This seems to be caused by bridges of dye molecules being formed between the polymer macromolecules. The direct dyes increased the sedimentation rate in Svedberg units from 1·6 in absence of dye to a rate of up to 6·2 as measured in a Phywe ultracentrifuge at 40,000 r.p.m. In one case, two maximum rates of sedimentation were observed, 2·4 and 6·2. The data show that, below a given dye concentration, bridges are not formed between the cellulose chains. C.O.C.

Continuous Piece-dyeing of Cotton
H. Waibel
Melliand Textilber., **39**, 1126–1128 (Oct. 1958)
Review of modern vat dyeing techniques.
S.M.J.

Use of the Turbinator in Textile Processing

I. L. Häusermann and E. Hablützel

Textil Praxis, **13**, 1167–1171 (Nov. 1958)

The principle of applying mechanical vibrations to assist the dyeing of textiles was first patented in 1876. During the last 80 years various improvements have taken place, and the application of the most recent development achieved by the firm of Benniger is described in this paper. The Turbinator consists of a stout tube, equipped with four blades, which is made to oscillate with a frequency of about 2,500/min., causing rapid vibrations of the fabric as it passes in the vicinity of the Turbinator. The incorporation of this mechanical vibrator is ideally suited for wet processing of fabrics in open width, e.g. on a jigger. It is claimed that processing time can be reduced by about a third with simultaneous saving of chemicals, that the resulting fabric is cleaner and more wettable, and that the majority of dyeings exhibit higher fastness to washing and rubbing.

L.A.T.

Adsorption at Organic Surfaces. V— Adsorption of Dyes and other Organic Solutes by Cellulose and Chitin

C. H. Giles and A. S. A. Hassan

J.S.D.C., **74**, 846–857 (Dec. 1958)

Adsorption experiments with cellulose and chitin and a variety of solutes, including non-ionic hydroxy compounds, aromatic sulphonic acids, and azo dyes, are described. In non-aqueous solvents the non-ionic compounds are adsorbed by both substrates, but in water they are adsorbed only by chitin. Almost all the sulphonated compounds are adsorbed by both substrates, but benzenesulphonic acid is not adsorbed by cellulose. It is concluded that cellulose adsorbs by hydrogen bond formation with the solute in absence of water, but in presence of water by van der Waals attraction alone. Chitin can adsorb by both of these mechanisms and also by ion exchange.

AUTHORS

Adsorption of Caustic Soda by Vegetable Fibres in Azoic Dyeing

J. Oxé

Textil Praxis, **13**, 1171–1172 (Nov. 1958)

The method adopted was to treat hanks of yarn in NaOH soln. of varying concentration (2, 4, 10, and 20 ml. of 32·5% NaOH per litre), with and without the addition of NaCl and alcohol. The excess of liquid was removed by wringing, leaving approx. 130% alkali soln. on the weight of yarn. After neutralisation with an acid, the amount of NaOH absorbed by various vegetable fibres was determined by back-titration of excess acid. The results indicate that at lower concn. of NaOH addition of NaCl or alcohol increases the absorption of alkali by cotton. Unbleached linen absorbs more NaOH than the bleached material. The absorption of NaOH by hemp is greater than that of coir and appreciably greater than that of jute.

L.A.T.

Uniform Results in the Pad Dyeing of Compact Fabrics with Indanthren Dyes

E. Schneider

Textil Praxis, **13**, 1044–1050 (Oct. 1958)

The author examines the causes of uneven dyeing along and across the piece in pad and jigger dyeing. Numerical examples are given which illustrate calculation of the composition of the pad liquor for satisfactory dyeing of raincoat poplins.

L.A.T.

Dyeing of Wool

Y. Nemoto and T. Imai

I— Level Dyeing of Wool by the pH-Control Process

Nagoyashi Kôgyô Kenkyûjo Kenkyû Hôkoku, **18**, 27–29 (1958): *Chem. Abs.*, **52**, 13268 (10 Aug. 1958)

Wool cloth (2 g.) was dyed in 200 g. 2% Eriochrome Black T (C.I. Mordant Black 11) solution at various pH and temperatures. The pH regulator used, pH, temperature, dyeing ratio after 5 and 120 min. and dyeing velocity were—

pH Regulator	pH	Temp. (°C.)	Dyeing Ratio (%) 5 min.	120 min.	Rate of Dyeing
Clark–Lubs buffer	1·5	100	83·2	95·6	0·44
		90	31·7	95·2	0·143
		80	21·0	92·6	0·108
		70	13·7	76·9	0·84
	3·5	100	56·8	92·1	0·257
		90	13·0	73·1	0·074
		80	7·3	64·2	0·059
		70	5·8	34·7	0·031
	5·5	100	22·8	77·5	0·100
		90	6·3	36·0	0·023
		80	4·3	20·2	0·023
		70	1·0	16·0	0·016
2% H_2SO_4	2·5–3·3	100	83·3	98·2	0·389
2% Acetic acid	3·8–4·2	100	75·0	98·3	0·375
5% NH_4 acetate	6·3–6·5	100	3·0	59·0	0·013
5% $(NH_4)_2HPO_4$	6·9–7·7	100	2·0	37·3	0·009
3% $(NH_4)_2SO_4$	5·4–6·7	100	14·0	70·0	0·063
15% NH_4Cl	5·4–5·5	100	34·7	91·2	0·155
20% Triacetin	5·0–5·1	100	47·0	95·3	0·211
10% Et lactate	5·1	100	42·1	95·1	0·188
100% Et malonate	5·1	100	41·8	95·1	0·187

II— Level Dyeing of Wool with Carbolan Dyes using Organic Esters as Assistants

Ibid., 30–32

Dyeing of wool cloth was carried out at 100°C. Dye, pH regulator, pH before dyeing, pH after dyeing, dyeing ratio after 60 and 120 min., dyeing velocity and whether dyeing was level (+) or not (•) were—

Dye	pH Regulator	pH Before Dyeing	pH After Dyeing	Dyeing Ratio (%) 1 hr.	2 hr.	Rate of Dyeing	Levelness
Carbolan Crimson BS (C.I. Acid Red 138)	2% Acetic acid	3·7	3·9	99·6	–	0·39	•
	0·2% Acetic acid	4·8	5·3	96·0	97·4	0·26	—
	(Control)	6·2	6·6	77·9	90·7	0·13	+
	2·4% Triacetin neutralised with NaOH	8·0	5·4	95·2	97·7	0·13	+
Carbolan Blue BS (C.I. Acid Blue 138)	2% Acetic acid	3·7	4·0	97·3	98·8	0·41	•
	0·2% Acetic acid	4·6	5·1	92·6	93·6	0·25	•
	(Control)	6·5	6·9	83·7	89·5	0·16	•
	0·054% Triacetin neutralised with NaOH	8·1	7·2	77·2	89·1	0·11	—
Carbolan Fawn R 150 (C.I. Acid Brown 49)	2% Acetic acid	3·6	3·9	99·5	–	0·27	•
	0·2% Acetic acid	4·2	4·7	94·6	96·8	0·19	—
	(Control)	6·0	6·4	63·7	77·0	0·10	+
	2·4% Triacetin neutralised with NaOH	6·0	5·3	74·8	91·4	0·11	+

C.O.C.

Reactive Dyes

J. Wegmann

Textil Praxis, **13**, 1056–1061 (Oct. 1958)

The author discusses in detail the chemical constitution of reactive dyes as a class with particular reference to the nature of solubilising groups, dye molecules, carriers of reactive groups, and reactive groups themselves. Technological aspects include application of reactive dyes by exhaustion and various padding techniques. There are 23 references to the literature.

L.A.T.

Reactive Dyes and the Pad-Roll Process
T. Vickerstaff
Z. ges. Textilind., **60**, 463–468 (5 June 1958)

The short liquor ratios used in the Pad–Roll process make this process particularly efficient for the application of reactive dyes of the Procion type. The best results are obtained at low temperatures, e.g. a dyeing time of 2 hr. at 20°c. in the presence of 10–20 g. Na_2CO_3 per litre.
J.C.F.

Spectral Dependence of the Fading of Dyed Textiles
R. E. Bedford
J.S.D.C., **75**, 37–44 (Jan. 1959)

A method for calculating the contributions of various spectral regions of light in causing fading is described, and results are given for three dyed samples. The materials were exposed to sunlight plus skylight, for which the spectral energy distribution is rather well known, behind a set of coloured filters. Fading was measured as an actual colour change by means of the Hunter colour-difference meter. A set of simultaneous equations is set up whose solution gives the relative efficiencies of the various regions in producing fading. A comparison of various equations to describe fading is also made.
AUTHOR

Ultraviolet Absorbers
R. A. Coleman and W. H. Peacock
Text. Research J., **28**, 784–791 (Sept. 1958)

Investigations made with 81 direct cotton dyeings and 76 wool dyeings on the effect on their light fastness of protection with a cellulose acetate film containing an ultraviolet absorber of the *o*-hydroxybenzophenone type are described, both for Fade-O-Meter and for direct sunlight exposures. Tensile strength tests carried out on the cotton materials showed that the loss of tensile strength was greatly reduced by screening out the ultraviolet light. Ultraviolet absorbers also retarded the yellowing of clear plastics and the spotting of polyvinyl chloride products. Although not substantive on cotton materials, some absorbers can be exhausted on to wool and man-made fibres to give a protective effect. S.B.D.

Fastness to Light of Fluorescent Brightening Agents
J. Pinte and P. Rochas
Ann. sci. textiles belges, **3**, 168–188 (1955): *Chem. Abs.*, **52**, 19503 (25 Nov. 1958)

In the visible spectrum wavelength has little effect on the degradation rate, but degradation proceeds rapidly under exposure to strong ultraviolet radiation. Yellowing of fabrics containing fluorescent brightening agents on exposure to light is caused primarily by disappearance of the fluorescence. In the case of nylon a slight real yellowing is added to the apparent and major yellowing caused by the loss of fluorescence. C.O.C.

Dyeing Mechanism of Neutral-dyeing Metal-complex Dyes. I— Adsorption Isotherms on Polyamide Fibres
G. Back and H. Zollinger
Helv. chim. Acta, **41**, 2242–2252 (Dec. 1958)

The dyeing equilibria of the 1:2 chromium complex of the dye 2-aminophenol-4-methyl-sulphonamide → 1-acetamido-7-naphthol between an aq. phosphate buffer and 6,6-nylon and acetylated 6,6-nylon have been determined at 100°c. Fixation of the dye by the fibres occurs by two independent mechanisms operating simultaneously — a salt-like linkage of the dye to the basic groups of the fibre which affords Langmuir adsorption isotherms, and a superposed solution mechanism. The value of the saturation adsorption by the salt-link mechanism calculated by extrapolation from the equilibrium data agrees with the amine end-group content of the fibres determined acidimetrically. H.H.H.

Aromatic Ammonium Azoic Diazo Components for Dyeing Polyester Fibres
E. Baier
Z. ges. Textilind., **60**, 283–284 (1958): *Chem. Abs.*, **52**, 17717 (25 Oct. 1958)

Azoic diazo components can be applied from an aqueous acidic solution of their hydrochlorides as at high temperatures these dissociate to yield the free base if the basicity of the latter is not too great. Examples of suitable bases are given. This method gives more level dyeings than does use of a dispersion of the base. It also makes possible application of an azoic diazo component and an azoic coupling component in the same bath. C.O.C.

Kinetics of Dyeing Acrylic Fibres with Basic Dyes
W. Beckmann
Melliand Textilber., **39**, 1121–1126 (Oct. 1958)

Investigations with a Dyeometer and a recording photometer have established that different dyes have similar highly temp.-dependent rates of dyeing, increasing by ca. 30% per degree near 100°c., corresponding to an activation energy of 72 kcal./mole. Increasing concn. markedly lowers rates of exhaustion owing to the limited number of acidic groups in the fibre. A similar lowering is caused by agitation gentler than that corresponding to a limiting rate. Dyeing curves for two concn. at one temp. enable others for different concn. and temp. to be constructed by interpolation from times of half-dyeing plotted linearly against concn. on a logarithmic scale and correcting for temp. The rate-determining concn. of a dye in a mixture, higher than the actual concn., is not equal to the total concn. of all dyes present, but obeys the relationship $C_{SA\,mix} = C_{SA} + P_A/P_B \times C_{SB}$ for a mixture of two dyes A and B, where C_{SA} and C_{SB} are concn. as fractions of the saturation concn., and P_A and P_B are "mixture parameters", related to diffusion coefficients, characteristic of the dyes, and independent of both amount and identity of other basic dyes in a mixture. Knowledge of these parameters, of saturation concn., and of times of 70% dyeing permits prediction of dyeing rates in mixtures, which are verified experimentally. Dyes with similar parameters dye at similar rates when mixed in all proportions. A retarder (Levegal PAN) behaves like a basic dye in these respects. S.M.J.

PATENT

Dyeing Cellulose Triacetate with Logwood (or its Derivatives (C.I. Natural Black 1 and 2)
Courtaulds *BP* 806,250

Satisfactory dyeings are obtained if a swelling agent is present in the dyebath but not in the mordanting bath. Thus cellulose triacetate cloth dyed with Hemasol AR (C.I. Natural Black 2) with addition of methyl ethyl ketone to the bath, rinsed and then treated with bichrome with NaCl in the bath, was given a full black of good fastness to light and washing. C.O.C.

Rayon Hank Dyeing (I p. 159)
Covalently Bound Azo Dyes (IV p. 161)
Pigmented Oil-in-Water Resin Emulsions for Printing and Dyeing (IX p. 175)

IX— PRINTING

White and Coloured Resists under Pigments
S. Matthes and G. Vogel
Melliand Textilber., **39**, 1251–1252 (Nov. 1958)

General principles reviewed include premature agglomeration of disperse resin binders by high electrolyte concn., and preventing acid-catalysed resin condensation with alkali. Two recipes are given. S.M.J.

Automatic Screen Printing Processes
A. Franken
Textil-Rund, **13**, 532–541 (Sept. 1958)

An illustrated review of several partly and fully mechanical processes. S.R.C.

Four-colour Printing. Screen process. Dye-bleach processes. Integral Masking. Iron Salt Sensitisers
K. M. Hornsby
Brit. J. Phot., **105**, 300–302 (1958)

Subbing Layer for Polystyrene Base. Stripping Films. Non-aqueous Coating Compositions. Anti-foggants and Stabilisers
Ibid., 302–301, 323, 334–335

Colour Coating Preparation. Line Colour Screens. Colour Translation System
Ibid., 368–369, 371

Colour Developing Agents. Masking Process. Duplicate Negative Process
Ibid., 416–417

Print-out Emulsion. Film Base Materials and Plasticisers. Subbing Hydrophobic Film. Image Protecting Layer
Ibid., 436–437, 439
Chem. Abs., **52**, 19629 (25 Nov. 1958)
Reviews of patents. C.O.C.

PATENTS

Pigmented Oil-in-water Resin Emulsions for Printing and Dyeing
Gy *BP* 806,571

Prints of good fastness to rubbing, laundering, and dry-cleaning are obtained by use of a viscous oil-in-water type colour concentrate comprising water, pigment, a dispersing agent for the pigment, a protective colloid and an emulsified resinous binder. This binder consists of pentaerythritol polyester alkyd resin and an organic solvent-soluble amino–aldehyde resin. The pentaerythritol resin is such that 50–100% by weight of its polyhydric alcohol component is a pentaerythritol and its acid component is a polybasic acid which contains phthalic acid and a polyunsaturated fatty acid. The pigment content is 4·5–40·0%. C.O.C.

Extenders for Oil-in-water Emulsion-type Printing Pastes
Sherwin-Williams Co. *USP* 2,844,547

An aqueous dispersion containing 2–8% of a galactomannan produced from seed endosperms of *Leguminosal* and 0·4–10·0% by weight of the dry galactomannan of a water-soluble ferrous salt has a pseudo plastic flow property. It can be used alone or with a hydrocarbon dispersed in it as an extender for oil-in-water emulsion type printing pastes. Such an extender has improved tolerance for electrolytes. C.O.C.

Photographs in a Variety of Colours on Paper, Cloth or Film
L. Chalkley *USP* 2,844,465

A photosensitive system is used which comprises at least two components both being necessary for the photochemical reaction and one being volatile. After the fully-formed image has been printed out it is fixed by evaporation of the volatile component. Thus water leaf paper is sensitised with a 0·5–1·0% solution of Crystal Violet cyanide in resorcinol dimethylether. It is then printed by an S4 sunlamp through a Corex D printing frame glass and an Indian ink drawing or tracing cloth until a sufficiently strong violet image (negative of the drawing) has been formed. The print is fixed by blowing air at 100°c. onto it until the odour of the resorcinol dimethyl ether has disappeared and exposure of a portion of the print to direct sunlight for 1 min. produces no appreciable image. C.O.C.

Mechanical Printing with Daylight-fluorescent Compositions
Switzer Bros. *USP* 2,845,023

Transfers for Application to Transparent Films
Dennison Manufacturing Co. *BP* 807,184

Developing and Fixing Exposed Silver Succindiamidedioxime
U.S. Secretary of the Army *USP* 2,841,494

A colloidal dispersion of silver succindiamidodioxime in a colloid carrier on exposure to light forms a latent image which is developed by steaming and fixed by treatment with ammonia. Sodium lauryl sulphate, sodium 2-ethyl hexene sulphonate, sodium tetradecyl sulphate, sodium dioctyl phosphate, and sodium heptadecyl sulphate can be used as sensitisers. C.O.C.

X—SIZING AND FINISHING

Evaluating Electrostatic Finishes
F. H. Steiger

Text. Research J., **28**, 721–733 (Sept. 1958)

Relations between charge, resistivity, and the concn. of antistatic finish are investigated. It is demonstrated that on a given fabric log (charge) is a linear function of log (surface resistivity), independent of the finish which produces the conductivity. S.B.D.

Finishing of Cellulose Triacetate Fabrics, with particular reference to Setting, Embossing, and Pleating
S. N. Bradshaw and A. S. Cluley

J.S.D.C., **74**, 842–846 (Dec. 1958)

Knowledge of thermal finishing of fabrics containing Tricel is reviewed and summarised, and an attempt is made to define the reasons for the different methods in use and to specify the sequence of operations in processing. AUTHORS

Drip-dry Resin Finishes for Cotton
J. D. Reid and R. M. Reinhardt

Modern Textiles, **39**, 61–68 (March 1958)

The authors review (27 references) resin finishes for cotton with special reference to urea, melamine, and cyclic ethyleneurea (CEU) resins and mention of polymeric glycol formals and diepoxides. The site of action of the resin on the fibre is considered, and typical details of the application of a CEU resin to cotton are given. The use of a CEU–acrylate–silicone resin for setting permanent creases in cotton garments is discussed. E.C.

Effect of Crease-resist Finishes on Crease Recovery—III
W. Wegener

Melliand Textilber., **39**, 1262–1268 (Nov. 1958)

A viscose filament taffeta containing 3, 7, and 14% of a dimethylolurea resin is creased in 7 directions with a weight of 1 kg. for 1 hr., and crease recovery angles are measured for times up to 1 hr. 23 graphs and 3 tables illustrate the results, which support the relationships previously found valid for another resin (*ibid.*, **39**, 671–678 (1958)) — $\alpha^\circ_{60} = 2{\cdot}69 + 1{\cdot}342\alpha^\circ_5$, where α° denotes the angle of crease recovery and the subscript the time in min. Time-independent measures of recovery are calculated. These increase from mean values of 29% for untreated fabric to 45% for the highest resin concn., whereas weight loss on abrasion only falls slightly. S.M.J.

Flameproofing of Textile Fabrics with particular reference to the Function of Antimony Compounds
N. J. Read and E. G. Heighway-Bury

J.S.D.C., **74**, 823–829 (Dec. 1958)

Some modern flameproofing processes are outlined, and the requirements of an ideal process discussed together with some methods of testing flameproofness and durability. The importance of ease of ignition as distinct from rate of burning as a fire hazard is referred to. The action of antimony oxide in association with hydrogen chloride liberated at ignition temperature is examined, and an experimental demonstration of the mechanism of the flameproofing effect of antimony chloride is described. The preparation, application, and performance of oil-in-water emulsions of plasticised polyvinyl chloride are discussed, and evidence of possible side-effects of the composition and antimony oxide is supplied. AUTHORS

Flame-resistant Finishing of Cellulosic Fabrics with Phosphorus-containing Resins
J. R. W. Perfect

J.S.D.C., **74**, 829–834 (Dec. 1958)

Methods of using phosphorus-containing compounds to produce durable flame-resistant finishes on cellulosic fabrics are discussed. Reference is made to finishes based on the use of amine–phosphate–formaldehyde resins, esterification methods, phosphorylamide compounds, tetrakishydroxymethylphosphonium chloride (THPC) resins, triallyl phosphates, and aziridinylphosphines. Particular mention is made of the commercially successful Proban finish, which is based on the use of THPC. A brief summary is given of suggested mechanisms for the flame- and glow-proofing action of phosphorus compounds. AUTHOR

Flameproofing of Textiles with special reference to Tetrakishydroxyphosphonium Chloride and other Phosphorus Compounds
J. R. W. Perfect

Textil-Rund., **13**, 464–469 (Aug. 1958): *Tinctoria*, **55**, 121–126 (April 1958)

A review of the properties and potentialities of the Proban finish. S.R.C.

Water-repellent Silicone Finishes
M. Kramer

Textil Praxis, **13**, 1050–1055 (Oct. 1958)

Recent developments in the application of silicones to textiles are discussed. Particular reference is made to a commercial product Perlit SI (FBy), which is not of methyl hydrogen polysiloxane type. The Bundesmann test (absorption and penetration) data have been tabulated for fabrics made from all major fibres treated under varying conditions. Results of spray tests are presented graphically. Advantages as well as drawbacks of proofing textiles with silicone products are outlined. L.A.T.

Water-repellent Finishes—Modern Use of Silicones
G. W. Madaras
J.S.D.C., **74**, 835–841 (Dec. 1958)

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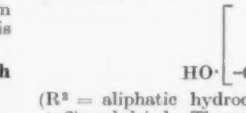

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S. A. Glikman, O. G. Efremova, I. K. Kosÿreva, and A. I. Somova
Zhur. priklad. khim., **31**, 1087–1091 (July 1958)

The thermal stability of ethylcellulose can be achieved by the removal from it of the low-mol.wt. oxidation decomposition products containing carboxyl groups, by ensuring neutralisation of these groups, or by purifying it from the compounds responsible for their formation. All the above conditions are satisfied when ethylcellulose is washed with a 2:1 mixture of acetone with water followed by the stabilisation with NaOH or when the unoxidised product is washed with boiling water, oxidised without stabilisation, and washed again. The ethylcellulose so produced can withstand a temp. of 130°C. for 15 hr. T.Z.W.

Ion-exchange Properties of Monocarboxycellulose
G. M. Skurikhina and V. I. Yur'ev
Zhur. priklad. khim., **31**, 931–937 (June 1958)

In ion exchange methods of estimating carboxyl groups in oxycellulose the pH of the equilibrium soln. decreases and therefore a low result is obtained. The pH decrease is directly proportional to the number of carboxyl groups. The potentiometric titration method for the estimation of carboxyl groups in a strongly oxidised cellulose with constant pH of the equilibrium soln. is given. The ion exchange capacity of the monocarboxyethylcellulose in relation to uni- and bivalent cations from 0·1 N. sol depends upon the pH of the equilibrium soln. and is approx. proportional to the number of carboxyl groups. T.Z.W.

PATENT

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DuP *USP* 2,842,510

The paper is impregnated with a thermosetting resinous precondensate, an oxyfluoroborate and, as plasticiser, a polyhydric alcohol or polyethylene glycol. The treated paper is not stiffer than untreated paper. C.O.C.

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Fibrous Protein and Polymer Structure—Certain Peculiarities
N. S. Andreeva and V. I. Iveronova
J. Polymer Sci., **31**, 257–262 (Sept. 1958)

A mathematical analysis is made of a system of parallel chain molecules which has a long-range order in the arrangement of the structural units along the chains, and a short-range order in the packing of the molecules. Meridional stripes between the arcs of the layer lines on X-ray diffraction photographs are interpreted as showing that in some parts of polymers there are regions where the parallel chains are arranged without any correlation with the packing of the units of neighbouring chains. P.T.S.

Denaturation of the Proteins of the Groundnut (*Arachis hypogaea*) with Guanidinium Salts, Metal Salts, and Urea
R. K. Williams and W. E. F. Naismith
J. Polymer Sci., **32**, 193–206 (Oct. 1958)

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Thermostable Ethylcellulose
S. A. Glikman, O. G. Efremova, I. K. Kosȳreva, and A. I. Somova
Zhur. priklad. khim., **31**, 1087–1091 (July 1958)

The thermal stability of ethylcellulose can be achieved by the removal from it of the low-mol.wt. oxidation decomposition products containing carboxyl groups, by ensuring neutralisation of these groups, or by purifying it from the compounds responsible for their formation. All the above conditions are satisfied when ethylcellulose is washed with a 2:1 mixture of acetone with water followed by the stabilisation with NaOH or when the unoxidised product is washed with boiling water, oxidised without stabilisation, and washed again. The ethylcellulose so produced can withstand a temp. of 130°C. for 15 hr. T.Z.W.

Ion-exchange Properties of Monocarboxycellulose
G. M. Skurikhina and V. I. Yur'ev
Zhur. priklad. khim., **31**, 931–937 (June 1958)

In ion exchange methods of estimating carboxyl groups in oxycellulose the pH of the equilibrium soln. decreases and therefore a low result is obtained. The pH decrease is directly proportional to the number of carboxyl groups. The potentiometric titration method for the estimation of carboxyl groups in a strongly oxidised cellulose with constant pH of the equilibrium soln. is given. The ion exchange capacity of the monocarboxyethylcellulose in relation to uni- and bivalent cations from 0·1 N. sol depends upon the pH of the equilibrium soln. and is approx. proportional to the number of carboxyl groups. T.Z.W.

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XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Fibrous Protein and Polymer Structure— Certain Peculiarities
N. S. Andreeva and V. I. Iveronova
J. Polymer Sci., **31**, 257–262 (Sept. 1958)

A mathematical analysis is made of a system of parallel chain molecules which has a long-range order in the arrangement of the structural units along the chains, and a short-range order in the packing of the molecules. Meridional stripes between the arcs of the layer lines on X-ray diffraction photographs are interpreted as showing that in some parts of polymers there are regions where the parallel chains are arranged without any correlation with the packing of the units of neighbouring chains. P.T.S.

Denaturation of the Proteins of the Groundnut (*Arachis hypogaea*) with Guanidinium Salts, Metal Salts, and Urea
R. K. Williams and W. E. F. Naismith
J. Polymer Sci., **32**, 193–206 (Oct. 1958)

Arachin Dissociation— Thermodynamic and Kinetic Study
R. K. Williams, W. E. F. Naismith, and C. F. H. Tipper
J. Polymer Sci., **31**, 35–44 (Aug. 1958)

The standard energy values have been calculated for the dissociation of arachin groundnut protein at various pH values, ionic strengths, temperatures, and protein concentrations. ΔH is small, probably because bonds (hydrogen bonds, salt-linkages) between the two parts before dissociation are replaced by bonds within each part after dissociation. ΔS is smaller than expected, probably because of a decrease in entropy due to alignment of water molecules on each part after dissociation. The dissociation constant is a function of the ratio, protein concentration/ionic strength. P.T.S.

XIII— RUBBER; RESINS; PLASTICS

Ziegler-type Polymerisations— Theoretical Study of the Mechanism
H. Uelzmann
J. Polymer Sci., **32**, 457–476 (Nov. 1958)

Effect of Physical State during the Electron Irradiation of Hydrocarbon Polymers
E. J. Lawton, J. S. Balwit, and R. S. Powell

I— Influence of Physical State on Reactions occurring in Polyethylene during and following the Irradiation
J. Polymer Sci., **32**, 257–275 (Nov. 1958)

Measurement are made of cross-linking, *trans*-vinylene formation, and hydrogen evolution using high- and low-density polyethylene irradiated at temperatures between − 170 and + 240°c. Cross-linking is mainly in the amorphous region of the polymer. *trans*-Vinylene unsaturation is produced with equal ease in the crystalline and amorphous regions. Radicals are trapped in the crystalline regions which can react with oxygen rather than form cross-linkages. Differences at different temperatures are mainly due to differences in crystallinity: between − 170 and + 34°c., for example, where crystallinity is nearly constant, hydrogen yield is constant.

II— Additional Experiments and Discussion pertaining to Trapped Radicals in Hydrocarbon Polymers
ibid., 277–290

Trapped radicals are investigated by means of infrared absorption at 5·8 μ., electron paramagnetic resonance, and gas uptake by the irradiated polymer. To produce trapped radicals at room temperature the polymer must be crystalline, glassy or highly cross-linked. Exposure to oxygen or ethylene dissipates trapped radicals. Heating above the glass transition temperature or the melting point also destroys them. Decay of radicals will take place in a vacuum at room temperature; it is arrested in liquid nitrogen. At room temperature radical life-time may be thousands of hours. In a vacuum decay is mainly due to delayed cross-linking. The mechanism of oxidative decay of radicals is discussed. Differences between the crystals of high- and low-density polyethylene cause different radical decay mechanisms. Irradiation can improve polymer properties, but trapped radicals can cause slow embrittlement. P.T.S.

Polyethylene Terephthalate— Dynamic Mechanical Properties
T. Kawaguchi
J. Polymer Sci., **32**, 417–424 (Nov. 1958)

The dynamical mechanical properties are measured between − 70 and + 310°c. at 100–200 cycles/sec. Two mechanical loss maxima are distinguished. Variations with degree of crystallinity, orientation, and water content are investigated. P.T.S.

Drawn Polyethylene Terephthalate Structure
W. J. Dulmage and A. L. Geddes
J. Polymer Sci., **31**, 499–512 (Sept. 1958)

Birefringence, infrared dichroism, and X-ray diffraction measurements. In uncrystallised samples, the plane of the molecules tends to be parallel to the plane of the film. At low extensions pronounced tipping of the crystallites occurs. Thus, the axis of the crystallites varies from a direction approximately perpendicular to the direction of stretch at low extension, to a direction almost parallel to the direction of stretch at high extensions. P.T.S.

Stretched Polyethylene— X-Ray Diffraction, Birefringence, and Infrared Dichroism
R. S. Stein

II— Generalised Crystal Orientation
J. Polymer Sci., **31**, 327–334 (Sept. 1958)

Two orientation functions may be derived quantitatively from X-ray diffraction data. These functions are sufficient completely to characterise the crystal contribution to birefringence and infrared dichroism. By subtracting the crystal contribution from the total birefringence or dichroism, the amorphous orientation may be characterised. The variations during thermal and mechanical treatment of the polymer can be studied.

III— Biaxial Orientation
Ibid., 335–343

Additional orientation functions can be determined from measurement of the variation in X-ray intensity with tilt of the polymer film. From these functions and those in the previous part, the crystal contribution to the birefringence and infrared dichroism or normal and tilted films can be calculated. P.T.S.

Polyethylene Terephthalate — Chromatographic Fractionation
S. D. Bruck
J. Polymer Sci., **32**, 519–521 (Nov. 1958)

Polyethylene terephthalate is dissolved in 20 : 80 trifluoroacetic acid–chloroform and poured on to an activated charcoal column. It is eluted with a 10 : 90 mixture of the same solvents. P.T.S.

High-polymer Infrared Spectra. VII— Polyacrylonitrile
C. Y. Liang and S. Krimm
J. Polymer Sci., **31**, 513–522 (Sept. 1958)

Infrared spectrum from 70 to 3200 cm.$^{-1}$. Polarised measurements on stretched and rolled specimens 350–3200 cm.$^{-1}$. Detailed assignments are proposed for all the absorption bands in the spectra. P.T.S.

Polyacrylonitrile— Molecular Weight
W. R. Krigbaum and A. M. Kotliar
J. Polymer Sci., **32**, 323–341 (Nov. 1958)

There is considerable disagreement between molecular weight data from different experimental methods. The procedure to obtain reliable osmotic pressure data depends on the method of preparation of the polymer, whether a whole polymer or a polymer fraction is being used, and the purity of the solvent. Difference between light-scattering and sedimentation-diffusion results is due to the omission of a hydrostatic pressure correction from sedimentation results, and the effects of depolarisation and fluorescence on light scattering. P.T.S.

Organic Polymers containing Phosphorus
V. V. Korshak
J. Polymer Sci., **31**, 319–326 (Sept. 1958)

A number of polyamides and polyesters derived from phosphorus-containing dicarboxylic acids have been synthesised. Polymers with aliphatic units in the chain are viscous oils or low-melting resins. Polymers with aromatic units in the chain are high-melting elastic solids. Comparison with analogous polymers not containing phosphorus shows that the introduction of phosphorus leads to greater flexibility. P.T.S.

Silicone Resin— Logarithmic Thermal Dissociation in Air
C. D. Doyle
J. Polymer Sci., **31**, 95–104 (Aug. 1958)

Rendering Polyvinyl Halide Resins Adherent to Glass (III p. 160)
Organophilic Hydrophobic Silicates, Silicas, Silicic Acids, and Hydrated Silicas— Fillers for Rubber and Plastics and Vulcanisers for Silicone Rubber (IV p. 167)
1958 Reviewed— Technical Developments in the Paint and Printing Ink Industries (V p. 169)
Tracing Cloth and the like (X p. 176)

XIV— ANALYSIS; TESTING; APPARATUS

Paper Chromatography of Disperse Dyes
C. D. Johnson and L. A. Telesz
J.S.D.C., **74**, 858–859 (Dec. 1958)

Polarographic Determination of Hydrogen Peroxide, Formaldehyde, and Acetaldehyde in Mixtures
S. Sandler and Y. Chung
Anal. Chem., **30**, 1252–1255 (July 1958)

A three-stage polarographic procedure is described in which hydrogen peroxide is determined in acid buffer soln., formaldehyde is determined in an alkaline soln. to which $TiCl_4$ is added to eliminate the interference of hydrogen peroxide, and acetaldehyde is determined in a similar soln. in the presence of dimedone, which reacts rapidly with formaldehyde. The relationship of diffusion current to concentration for each pure substance is the only calibration requirement. L.T.W.

Submicro Methods for the Analysis of Organic Compounds. V— Determination of Sulphur
R. Belcher, R. L. Bhasin, R. A. Shah, and T. S. West
J.C.S., 4054–4056 (Nov. 1958)

Sulphur in org. cpd. is determined by decomposing ca. 50-μg. samples with fuming HNO_3 (sp.gr. 1·5) and anhyd. $BaCl_2$ at 275–280°c. The pptd. $BaSO_4$ is centrifuged off, washed, and dissolved in excess of ammoniacal 0·02 M. disodium ethylenediaminetetra-acetate (E.D.T.A.), and the sulphur determined indirectly by titration of the excess of E.D.T.A. with standard 0·01 M.-$MgCl_2$ (screened Solochrome Black T indicator). Average results for any one cpd. are within 0·20%; no result is likely to exceed by 0·4% the absolute sulphur content. H.H.H.

Characterisation of Thiols by means of their *pp'*-Nitrophenylazobenzoyl Derivatives
El S. Amin
J.C.S., 4769–4770 (Dec. 1958)

pp'-Nitrophenylazobenzoyl chloride, already used for the identification and separation of alcohols and amines, is now applied to thiols, and the thiol-esters, $NO_2{\cdot}C_6H_4{\cdot}N{:}N{\cdot}C_6H_4{\cdot}CO{\cdot}SR$ (where R = CH_3, C_2H_5, *n*-C_3H_7, *n*-C_4H_9, *n*-C_5H_{11}, *n*-C_6H_{13}, *n*-C_7H_{15}, *n*-C_8H_{19}, C_6H_5, *p*-$C_6H_4{\cdot}CH_3$, and $CH_2{\cdot}C_6H_5$), which have sharp m.p. in a convenient range, in contrast to other reagents, and crystallise well, are now described. The esters are brightly coloured which assist in chromatographic separations, some examples of which are recorded. H.H.H.

Separation of Sulphate, Sulphite, Thiosulphate, and Sulphide with Anion-exchange Resins
A. Iguchi
Bull. Chem. Soc. Japan, **31**, 600–605 (July 1958)

Sulphate, sulphite, and thiosulphate may be separated by means of an anion-exchange resin (e.g. polystyrene-divinylbenzene quaternary amine). Elution with 0·1 M.-$NaNO_3$ adjusted to pH 9·7 with NH_4OH removes first sulphite followed by sulphate. Thiosulphate is eluted last with 1·0 M.-$NaNO_3$. If sulphide is present the first eluant is 0·1 M.-NH_4NO_3 adjusted to pH 9·7 with NH_4OH plus 30% (by vol.) of acetone. This removes successively sulphite and sulphide. Sulphate is then eluted with 0·1 M.-$NaNO_3$ followed by thiosulphate with 1·0 M.-$NaNO_3$. Changes in the distribution coefficients of the ions in varying eluants are given. A.J.

Substituted Benzidines and Related Compounds as Reagents in Analytical Chemistry. XV— Some New 3,3'-Disubstituted Naphthidines
R. Belcher, S. J. Lyle, and W. I. Stephen
J.C.S., 4454–4458 (Dec. 1958)

The redox properties of a range of substituted naphthidines having in the 3- and 3'-positions the groups C_2H_5, *n*-C_3H_7, *i*-C_3H_7, $O{\cdot}CH_3$, C_6H_5, NO_2, NH_2, and SO_3H, are reported, and their properties of interest in analytical processes are described. Spectrophotometric data for the oxidised naphthidines, sensitivities towards oxidants, and transition potentials are given for the reactive cpd. Their behaviour as indicators is given for several conventional titrations. H.H.H.

Moisture Content of Textiles determined by Infrared Reflectance
K. Hoffmann, L. Schlamp, and F. Gläser
Melliand Textilber., **39**, 1157–1160 (Oct. 1958)

Infrared reflectance curves for wool, linen, paper, and leather alter with moisture content substantially in accord with the Kubelka–Munk function. The smooth curves given by plotting weight gain against the ratio of reflectance at 1·96 μ. (lowered by water) to that at 1·7 μ. (unaffected by water) enable moisture contents to be determined with an accuracy of $\pm$ 0·2%. S.M.J.

Colorimetric Determination of Bivalent Copper with Sodium Alizarin-3-sulphonate
A. Mukherji and A. K. Dey
Bull. Chem. Soc. Japan, **31**, 521–524 (July 1958)

Copper (II) forms a sol. 1:1 complex with 1:2-dihydroxyanthraquinone-3-sulphonic acid, which has λ_{max} ca. 500 mμ. and is suitable for colorimetric determination of Cu^{2+}. The colour is stable at 20–30°c. and pH 3·2–7·5. Sensitivity is 6·4 p.p.m. of Cu^{2+}. Tolerances of many other ions are listed. A.J.

Chromatographic Analysis of Dye Intermediates
S. Hoffman, V. Paul, and B. Iacob
Rev. chim. (Bucharest), **9**, 44–46 (1958):
Chem. Abs., **52**, 19717 (25 Nov. 1958)

A circular filter paper (Schleicher–Schüll No. 589) technique was used to separate 1-amino-8-naphthol-3:6-disulphonic acid, 1-naphthylamine-3:6:8-trisulphonic acid, 1:8-dihydroxynaphthalene-3:6-disulphonic acid from a 10% NaCl solution (R_f 0·6, 0·8, 0·6, and 0·8 respectively). The acids were identified by their fluorescence. To separate the 1-naphthylamine-4-sulphonic and 1-naphthylamine-5-sulphonic acids, the ascending method was used, from 10% brine as solvent, in an atmosphere saturated with the mixture butanol–acetic acid–water (4:1:5). The fluorescent spots separated at R_f 0·7 and 0·79. C.O.C.

Paper Electrophoresis of Leuco Sulphur Dyes
H. Tajiri
J.S.D.C., **74**, 860–862 (Dec. 1958)

Separation and Identification by Paper Chromatography of Food Dyes
G. Serini
Chimica (Milan), **34**, 95–96 (1958)

I— The following dyes can be detected in extracts from foods by using circular chromatography and butanol–pyridine–water (6:4:3) as solvent; C.I. Food Black 1 (R_f 0·26), C.I. Food Yellow 4 (0·29), C.I. Food Red 9 (0·32), C.I. Food Blue 1 (0·38), C.I. Food Yellow 13 (0·40), C.I. Food Yellow 3 (0·52), Ponceau GR (0·58), Azorubine (0·58) and C.I. Food Red 14 (0·82).

II— A New Chromatographic Technique on Paper Strips
Ibid., 144–145

The above dyes when separated by chromatography on a long paper strip yielded R_f values different from the above but in the same order.

Ibid., 197–200:
Chem. Abs., **52**, 20712 (25 Nov. 1958)

III— C.I. Food Red 4 (0·46) and C.I. Food Blue 3 (0·56) were separated chromatographically on paper strips, the infrared spectra of the separated dyes on paper are given. C.O.C.

Detection of Synthetic Dyes in Edible Oils or Butter
L. Guglielmi
Olii minerali, grassi e saponi, colori e vernici, **35**, 4–5 (1958):
Chem. Abs., **52**, 20745 (25 Nov. 1958)

Synthetic dyes can be detected in edible oils or butter by the reactions of Villavecchia–Fabris and Isidoro–Parolini (reaction with furfural). A mixture of 3 c.c. oil, 3 c.c. petroleum ether (b.p. 50–70°c.) and 3 c.c. of reagent (10 g. conc. HCl, 50 g. glacial acetic acid and 5 g. water) is shaken, if a synthetic dye is present then the acid layer becomes pink or red. Butter is similarly tested using as the reagent 40 g. conc. H_2SO_4, 90 g. glacial acetic acid and 10 g. water, presence of synthetic dyes is indicated by the acid layer becoming violet. C.O.C.

Determining the Sulphur Content of Wool
M. V. Glynn
Text. Research J., **28**, 744–746 (Sept. 1958)

The determination of the sulphur content of wool by digestion in boiling soln. of equal parts of 72% perchloric acid and conc. nitric acid, and subsequent pptn. of the resulting sulphate as barium sulphate for gravimetric estimation is described. The results are comparable with those obtained by standard microanalytical methods both for virgin wools and for wools which have been modified to contain the more resistant monosulphide linkage of lanthionine in place of the disulphide linkage of cystine. S.B.D.

Colorimetric Determination of Rutin (C.I. 75730)
V. Bilińska

Acta Polon. Pharm., **15**, 123–128 (1958): *Chem. Abs.*, **52**, 20893 (25 Nov. 1958)

Alcoholic solutions of rutin were treated with $TiOSO_4$ and the colour produced measured photometrically at 533 and 541 mμ. The results agreed completely with the Lambert–Beer Law. The method is useful for determining rutin in commercial products. C.O.C.

Moisture Content Determination by Drying
H. Sommer and F. Winkler

Melliand Textilber., **39**, 1138–1141 (Oct. 1958)

The residual moisture of textiles dried at elevated temp. still varies appreciably with the humidity of the drying air. This is substantiated quantitatively, and 16 references concerning drying and the establishment of commercial weights are discussed. The authors advocate standardisation of the moisture content at 10 ± 3 g./kg. of dry air. S.M.J.

Differentiation of Ripe and Unripe or Dead Cotton
P.-A. Koch

Z. ges. Textilind., **60**, 457–462 (5 June 1958)

A concise summary, with tables and photomicrographs, of methods for determining the degree of maturity of cotton fibres. 60 references. J.C.F.

Isolation and Identification of Fumaric Acid in Raw Cotton
L. E. Gregory and G. V. Merola

Text. Research J., **28**, 813–814 (Sept. 1958)

Raw cotton contains an average of 0·84% organic acids, made up of malic, citric, and oxalic acids together with unknown acids. The acids were identified by eluting centrifuged alcoholic extracts of raw cotton from a column with 6 N. formic acid, and then evaporating to dryness before separating by paper chromatography. The resulting chromatograms gave spots with R_f values identical to those given by authentic samples of malic, citric, and fumaric acids. Isolation of the fumaric acid was achieved by extracting 100 g. of raw cotton with 80% alcohol and eluting through a small column with 6 N. formic acid, using paper chromatography to check the purity of each small fraction collected. Those showing fumaric acid were re-eluted, and the fractions free from citric acid then evaporated to dryness. The yield on 100 g. cotton was 56 mg. and the m.p. 280°C. against 285°C. for the authentic acid. S.B.D.

Quantitative Separation of Wool and Regenerated Cellulose Fibres with Sodium Hypochlorite
M. Kehren and C. Maurer

Z. ges. Textilind., **60**, 766–769 (5 Sept.); 810–813 (19 Sept. 1958)

0·4–0·5 g. of the blended material, previously extracted with ether, is dried for 3 hr. at 105°C., and the dry weight determined. The sample is treated at room temp. with 60 ml. of 1 N.-NaOCl (35·46 g. active Cl per litre, with the addition of 5 g. NaOH per litre) for 15 min. with vigorous stirring. The residue of cellulosic fibre is collected on a No. 2 sinter and washed with water, 0·5% acetic acid, and water again. The residue is dried and weighed, and the weight is multiplied by 1·007, a correction factor determined by the average weight loss of a large number of viscose and cuprammonium rayons. A minimum of three replicates per determination is recommended. J.C.F.

Determination of Damage to Wool Fibres
J. C. Brown

J.S.D.C., **75**, 11–21 (Jan. 1959)

Tests for chemical damage to the wool fibre are classified according to the type of modification measured. Microscopical and staining tests are discussed, and a new test for alkaline damage is described. The swelling of wool fibres in aqueous buffer solutions has been accurately measured using a centrifuge technique under easily reproducible controlled conditions. Rupture of disulphide bonds by alkaline modification leads to a rapid increase in percentage swelling, which can be used as a measure of damage. A slight proportional decrease in swelling capacity results from dyeing, and an interesting distinction has been made between neutral-dyeing metal-complex dyes and other types of wool dyes. Chrome dyeings possess greater resistance to alkaline attack than undyed wools. AUTHOR

Synthetic Fibres— Reaction with Zinc Chloroiodide
M. Stratmann

Z. ges. Textilind., **60**, 450–456 (5 June 1958)

The microscopic behaviour of the principal types of synthetic fibres with zinc chloroiodide solution is described. Nylon or Perlon may be distinguished from Rilsan, and characteristic behaviour is shown by fibres of polyvinyl acetate, polyacrylonitrile, polyvinylidene cyanide (Darvan) and the mixed polymer Zefran. Polyester and polyvinyl chloride fibres do not react. There are 19 photomicrographs. J.C.F.

Two Tests for Detecting Nitriles and Amides
S. Trofimenko and J. W. Sease

Anal. Chem., **30**, 1432–1434 (Aug. 1958)

Two tests are described which detect and readily differentiate nitriles and amides in which there are none, one, or two substituents on the amide nitrogen. One test depends on detection of ammonia or amine with methanolic copper sulphate after pyrolysis with soda lime, and the other on formation of an organomercury compound with mercuric chloride. L.T.W.

Fibre Identification by Chemical Reactions
P.-A. Koch and M. Stratmann

Melliand Textilber., **39**, 1141–1145 (Oct. 1958)

Previous solubility schemes do not always accommodate the newest synthetic fibres such as acrylic copolymers and 11-nylon, or differentiate them sufficiently from closely related materials. Three tables, up to date to mid-1958, show respectively— the behaviour of 27 fibres with 24 solvents, both hot and cold; "type-reactions", each combination of two or three being characteristic of a particular class of fibre; and a complete solubility scheme for qualitative fibre analysis utilising 13 solvents. Clear instructions are given, and allowance is made for borderline cases, the interpretation of which might differ with the observer. 13 references to recent work are cited. S.M.J.

Description and Measurement of White Surfaces (I.S.C.C. 1953 Subcommittee Report)
R. S. Hunter

J. Opt. Soc. Amer., **48**, 597–605 (Sept. 1958)

Problems in colour technology of white surfaces are considered, and a survey of instruments and methods is given. Instruments have little, if any, advantage over visual inspection, since, although lightness can be measured with a precision exceeding that of the eye, horizontal differences in the colour solid cannot be measured precisely. However, instruments furnish more permanent records than do mere visual inspections. It is suggested that the textile industry might follow the paper industry in making use of blue reflectance, especially if additionally evaluated with an ultraviolet-absorbing filter over the illuminant. It is hoped that further work with direct-reading instruments like the Cotton Colorimeter (Nickerson, Hunter, and Powell, *ibid.*, **40**, 446 (1950) and with whiteness formulae may lead to more widespread familiarity with, and acceptance of, single-number whiteness rankings from tristimulus data. R.B.B.

Measurement of Colour Fastness to Steam Pleating
Society of Dyers and Colourists Pleating Fastness Subcommittee

J.S.D.C., **75**, 31–37 (Jan. 1959)

The Subcommittee has studied the problem of testing the fastness of coloured textiles to steam pleating processes and has devised a suitable test method. There is good correlation between the results of the test and those produced in practical pleating. A simple piece of apparatus has been designed to maintain close and even contact between the test pattern and adjacent undyed fabrics. Any staining of these adjacent fabrics takes place uniformly, and reliable assessment of the degree of staining can be made. A range of steaming conditions has been chosen to cover all fibres. The test has been adopted as an I.S.O. Tentative Test, the only modification being the addition of two extra steaming conditions. AUTHOR

Analysis, Composition and Purification of Commercial Copper Phthalocyanine (C.I. 74160) (IV p. 163)
Paper-chromatographic Study of Numerous Acid Dyes. II (IV p. 163)
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Evaluating Electrostatic Finishes (X p. 175)

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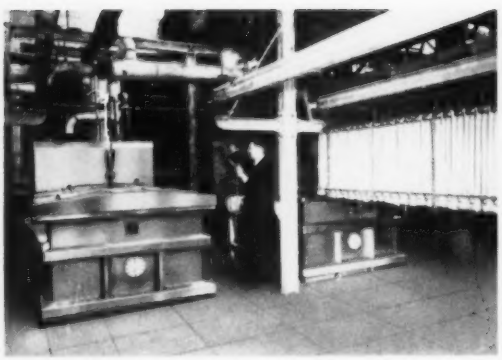

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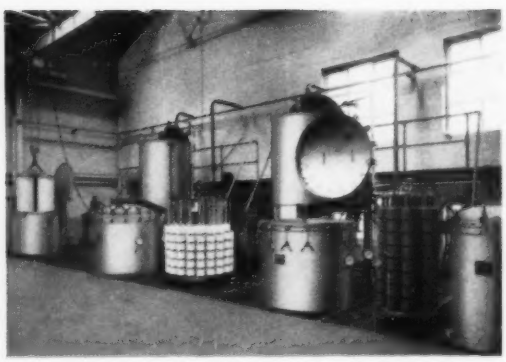

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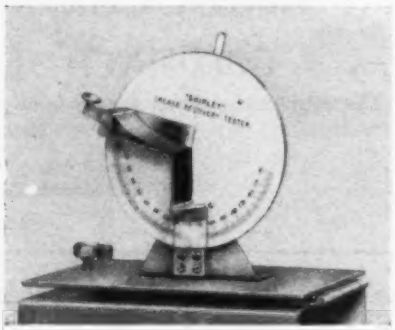

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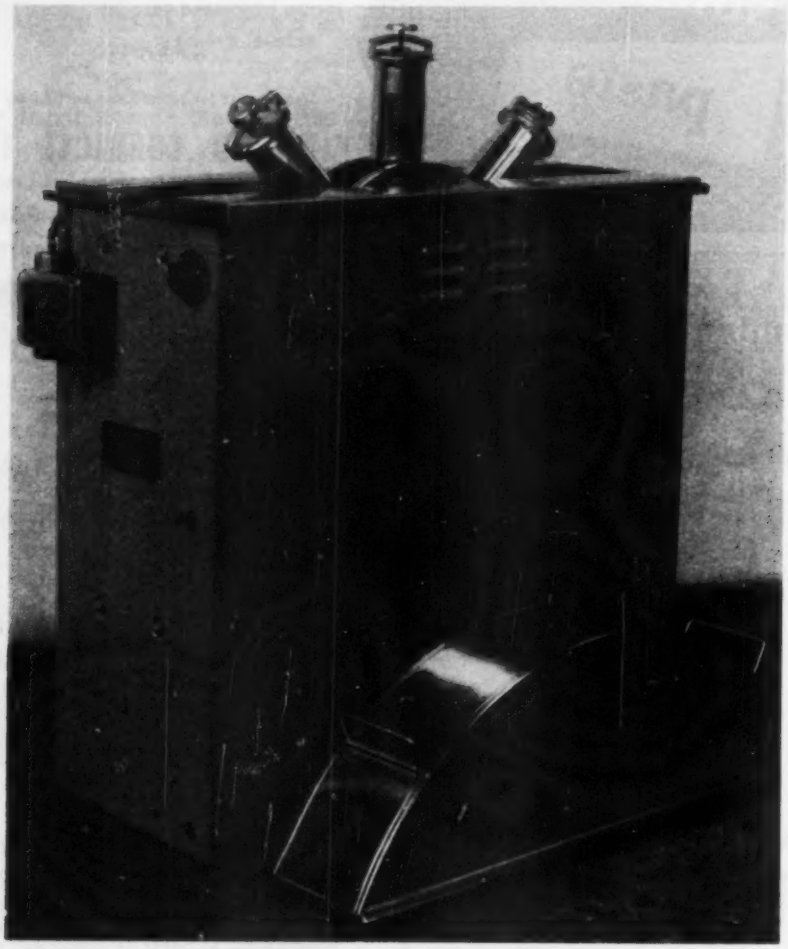

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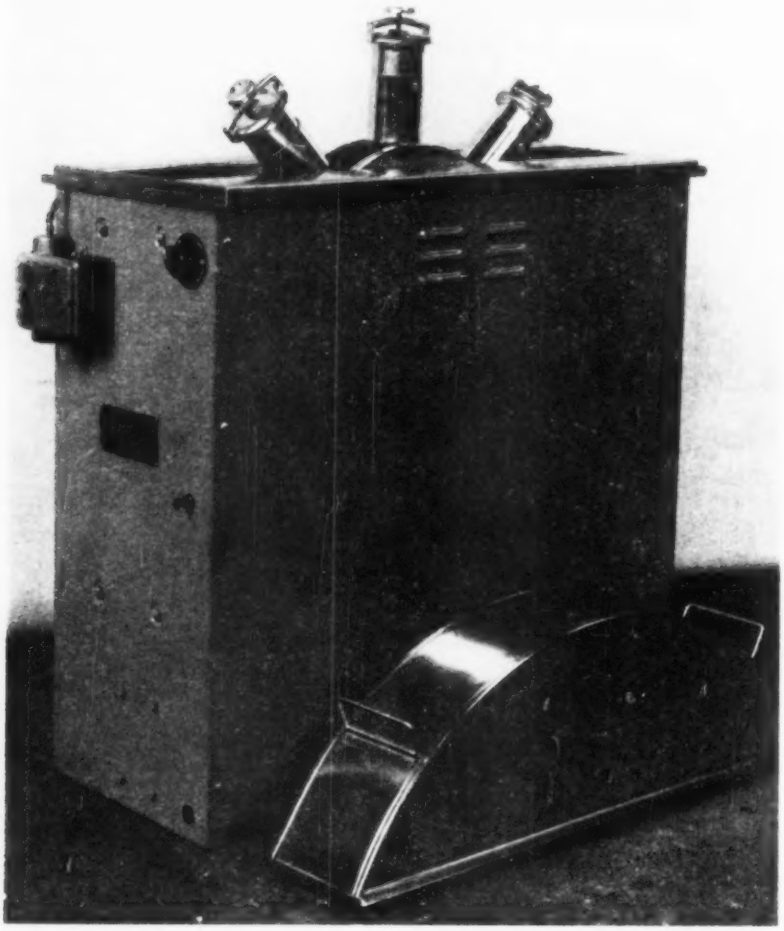

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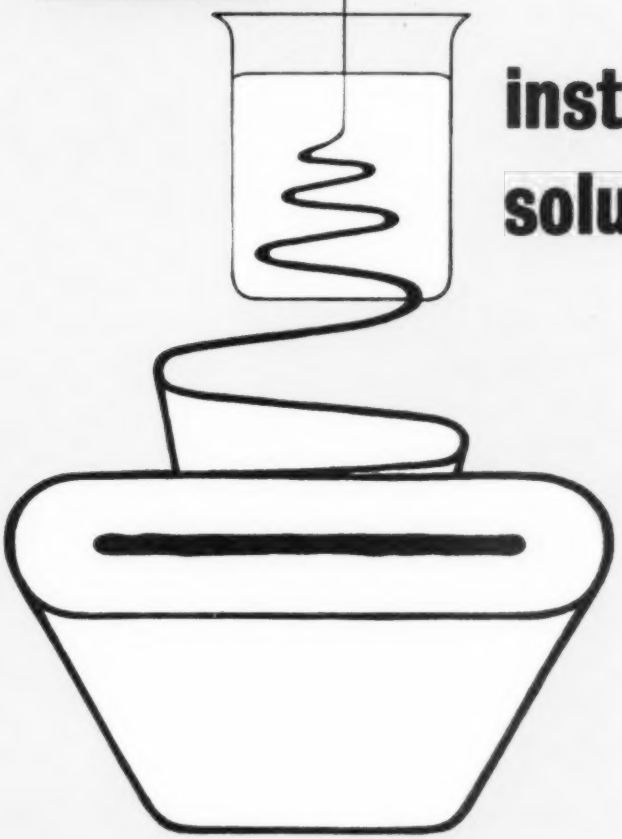

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